REMEDIAL ACTION WORK PLAN FOR SITES SS003 AND SS004 OIL SATURATED AREA AND HAZARDOUS WASTE DRUM STORAGE AREA KANSAS CITY MO

10/1/1991

BURNS & MCDONNELL

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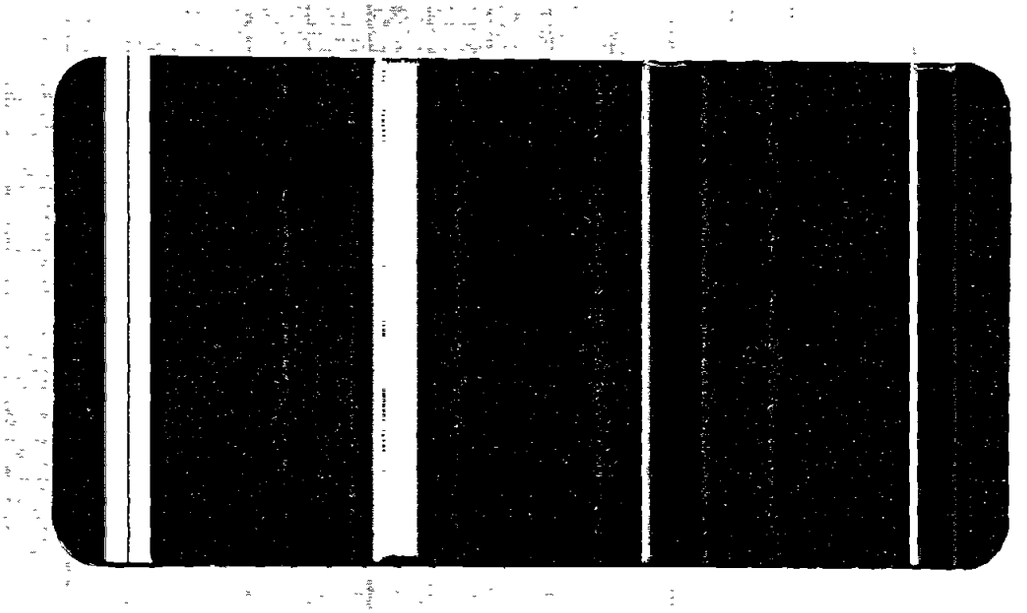

CONTRACT NO: F23608-91-D0020-5002
RG 91-0045, REMEDIAL ACTION, SITES SS03/SS04
OIL SATURATED AREA AND
HAZARDOUS WASTE DRUM STORAGE AREA
RICHARDS-GEBAUR AIR FORCE BASE, MISSOURI

WORK PLAN

October 1991

91-804-3-008

Burns & McDonnell
Engineers-Architects-Consultants
Kansas City, Missouri

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LIST OF ABBREVIATIONS

ACGIH	American Conference of Governmental Industrial Hygienists
ANSI	American National Standards Institute
ARAR	Applicable or Relevant and Appropriate Requirements
ASTM	American Society for Testing and Materials
BNAs	Base Neutral/Acids Extractables
CBC	Complete Blood Count
CERCLA	Comprehensive Environmental Response, Compensation and Liability Act
CFR	Code of Federal Regulations
CME	Central Mines and Equipment
CPR	Cardiopulmonary Resuscitation
DCP	Data Collection Plan
DQO	Data Quality Objective
EPA	Environmental Protection Agency
FID	Flame Ionization Detector
FSM	Field Site Manager
GGTP	Gamma Glutamyl Transpeptidase - Liver Enzyme Test
GOC	Ground Operations Center
HCN	Hydrogen Cyanide
HNU	HNU Instrument Company
HPLC	High-Performance-Liquid-Chromographic-Grade Water
HSO	Health and Safety Officer
kg	kilogram
LEL	Lower Explosive Limit
MDNR	Missouri Department of Natural Resources
MSA	Mine Safety Appliances
N	North
NIOSH	National Institute for Occupational Safety and Health
OSHA	Occupational Health and Safety Administration
OVM	Organic Vapor Monitor
PAH	Polynuclear Aromatic Hydrocarbon
PARCC	Precision, Accuracy, Representativeness, Comparability, and Completeness
PID	Photoionization Detector
PM	Project Manager
QAPP	Quality Assurance Project Plan
RCRA	Resource Conservation and Recovery Act
RFI	RCRA Facility Investigation
RGAFB	Richards-Gebaur Air Force Base
RPD	Relative Percent Difference
S	South
SHSP	Site Health and Safety Plan
SHSS	Site Health and Safety Supervisor
SI	Site Inspection
SMAC	Blood Chemistry Tests
SWMU	Solid Waste Management Unit
TIP	Total Ionizables Present
TLV	Threshold Limit Value
USGS	United States Geological Survey
VOC	Volatile Organic Compound

WBGT	Wet Bulb Globe Temperature
°C	Degrees Celsius
°F	Degrees Fahrenheit

* * * * *

1.0 INTRODUCTION

1.0 INTRODUCTION

1.1 SCOPE OF WORK

This Work Plan has been prepared by Burns & McDonnell Engineering Company for the activities associated with the removal of petroleum contaminated soil from two sites located on the Richards-Gebaur Air Force Base (RGAFB) (see Figure 1). Approximately 20 cubic yards of soil will be removed from the Oil Saturated Area, (Site SS03); and approximately 23 cubic yards of soil will be removed from the Hazardous Waste Drum Storage Site (Site SS04). The locations of these two sites are shown on Figure 2.

Included in this Work Plan are a Field Work Plan, a Sampling and Analysis Plan, a Quality Assurance Project Plan and a Site Health and Safety Plan. Field activities for the remediation will include removing and stockpiling petroleum and lead contaminated soil from the two sites; confirmation soil sampling of the exposed subgrade; soil sampling to determine the proper disposal method for the excavated soil; and the backfilling and resurfacing of the two excavated areas. All soil samples will be analyzed for lead and total petroleum hydrocarbons (TPH).

1.2 SITE LOCATION

RGAFB is located in west-central Missouri, about 2.6 miles east of the Kansas state line, as shown on Figure 1. The base is almost equally divided by the Jackson and Cass County Line, which runs east-west through the middle of the base. In Cass County, the base is bounded by the city of Belton on the east and south and in Jackson County, the base is bounded by Kansas City. Downtown Kansas City is about 18 miles to the north. Grandview is about 3 miles to the northeast and Belton is about 3 miles to the southeast. The main access to the base is off of U.S. Highway 71 (Reference 1). The Oil Saturated Area (Site SS03) is located to the southwest of Building 704 in the central part of the RGAFB (see Figure 2). The Hazardous Waste Drum Storage Site (Site SS04) is located to the southwest of Building 923 and to the northwest of Building 924 in the central part of RGAFB (see Figure 2).

1.3 SITE HISTORY

1.3.1 Oil Saturated Area - Site SS03

Site SS03 was originally used to store waste engine oils and waste transmission fluids originating from base vehicle maintenance in Building 704. Spillage due to waste transfer, overfilled drums, and leaking drums has discolored the surface soils in this area. In 1980, a gravel cover was placed over the area to stabilize the oil-saturated soils. In 1986, approximately one-half of the site was paved with asphalt, and then overlain with asphalt in 1989 (Reference 2).

Since 1983, Site SS03 has been the subject of studies which confirmed that site contamination is limited to petroleum-based organics and lead (Reference 2). In October 1986, the site was tested for purgeable aromatics, purgeable halocarbons, total petroleum hydrocarbons, and lead (see Figure 3). In August 1989, this site was tested for purgeable aromatics, purgeable halocarbons, base neutrals and acid extractables, and total metals (see Figure 4) (Reference 2). A summary of the analytical results for this site is compiled in Table 1. The volume of contaminated soil at the Oil Saturated Area is

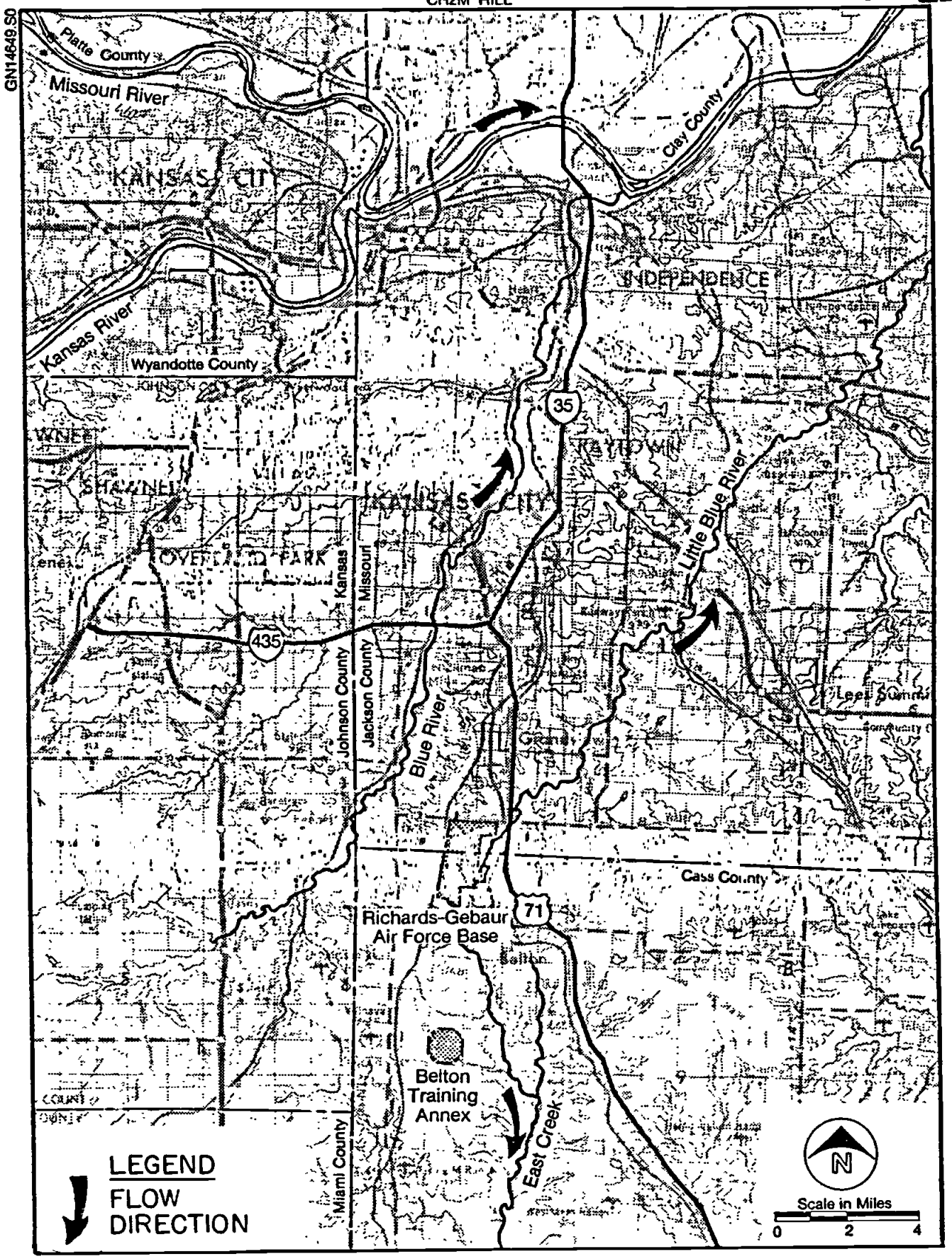


FIGURE 1
RICHARDS-GEBAUR AIR FORCE BASE LOCATION MAP

FROM: INSTALLATION RESTORATION
PROGRAM RECORDS SEARCH
CH2M HILL, MARCH 1983

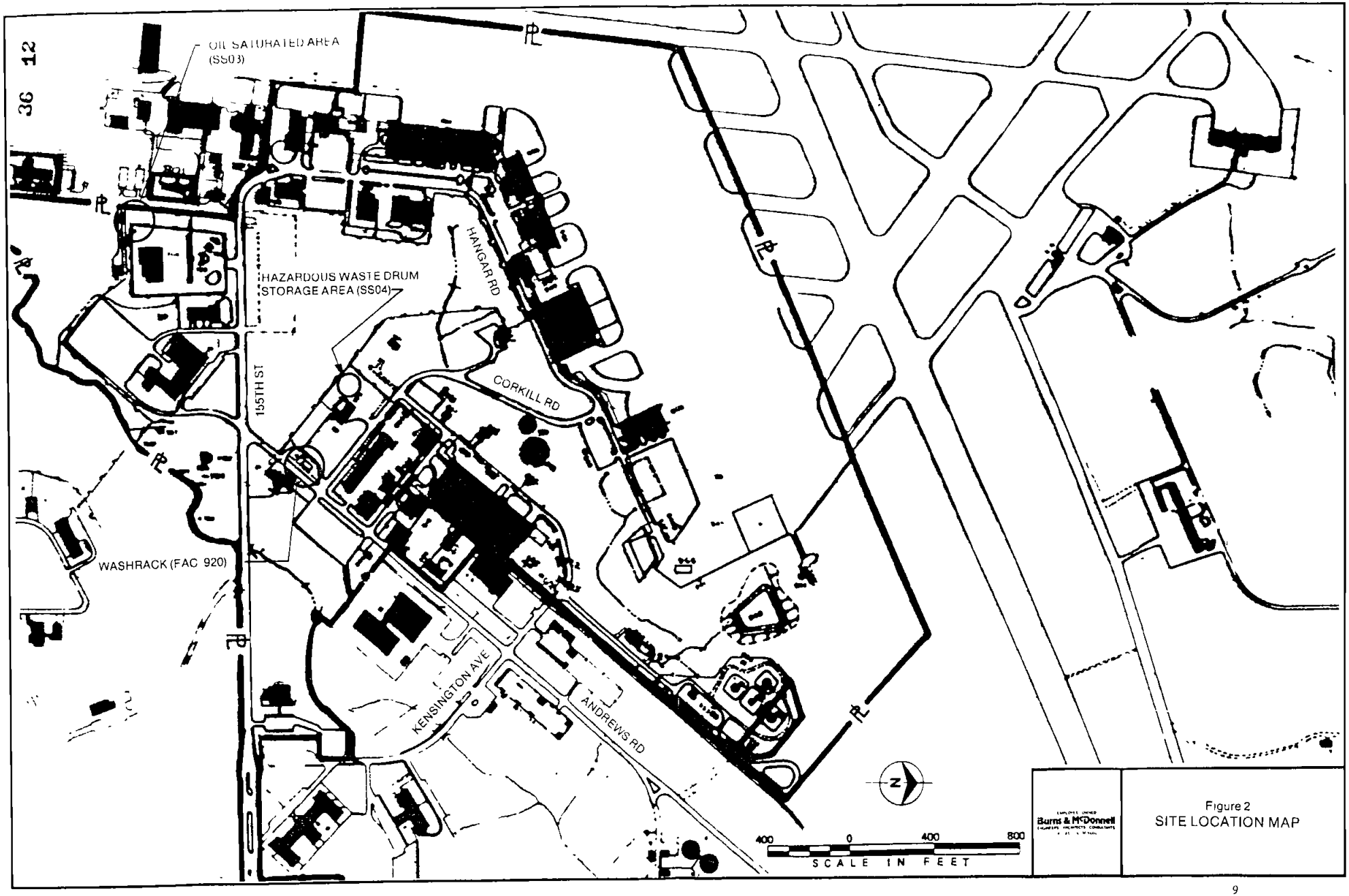
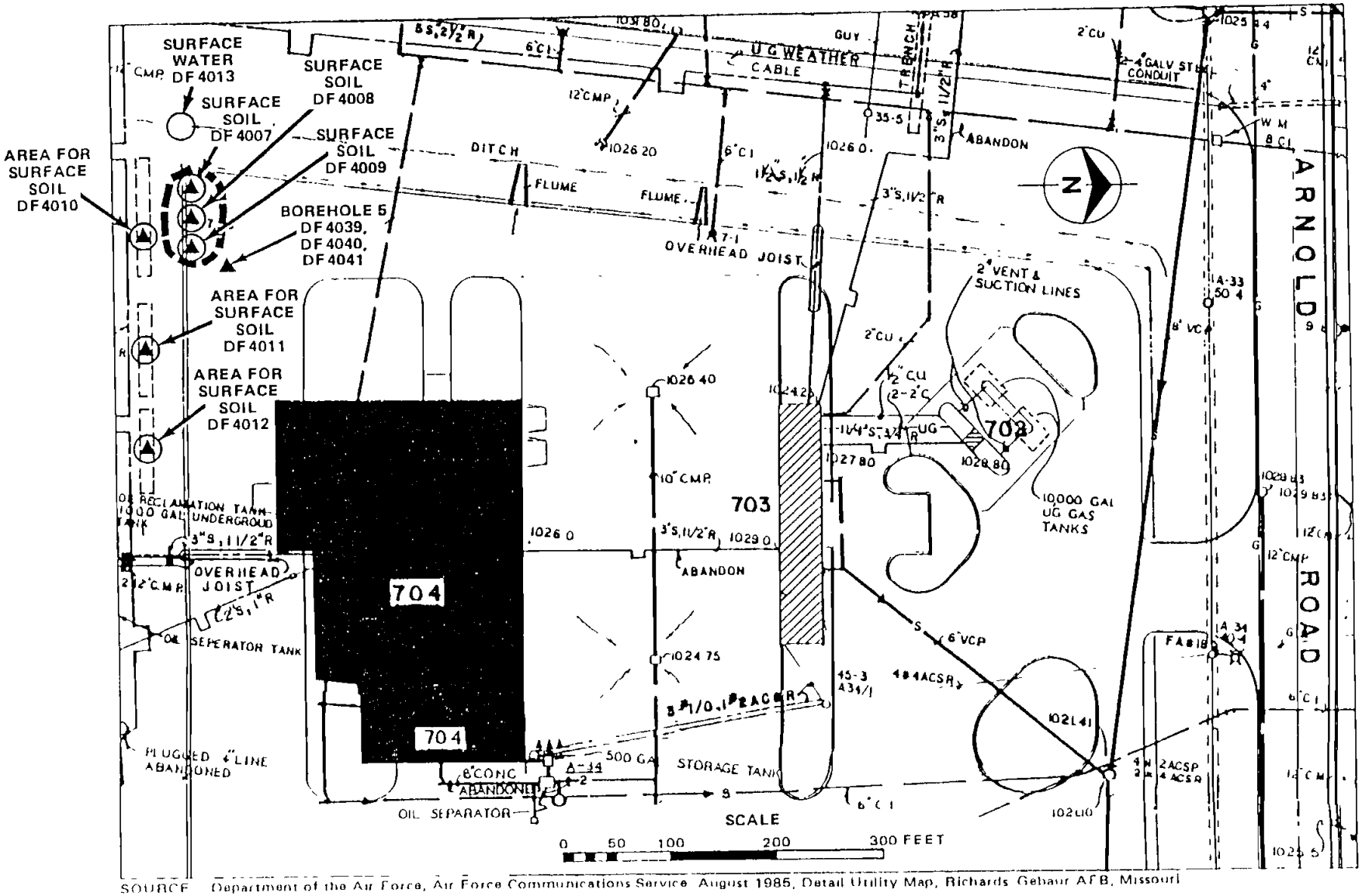


Figure 2
SITE LOCATION MAP



SOURCE: Department of the Air Force, Air Force Communications Service, August 1985, Detail Utility Map, Richards Gebaur AFB, Missouri

KEY:
- Site Boundary
- Surface Runoff Direction
- Subsurface Soil Sampling Location
- Surface Soil Sampling Location
- Groundwater Sampling Location
- Surface Water Sampling Location

FROM "INSTALLATION RESTORATION PROGRAM, PHASE II, CONFIRMATION/QUALIFICATION STAGE 2," ECOLOGY AND ENVIRONMENT, INC JULY 1988

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Figure 3
OCTOBER 1986
SAMPLE LOCATIONS,
OIL SATURATED AREA
SITE SS03

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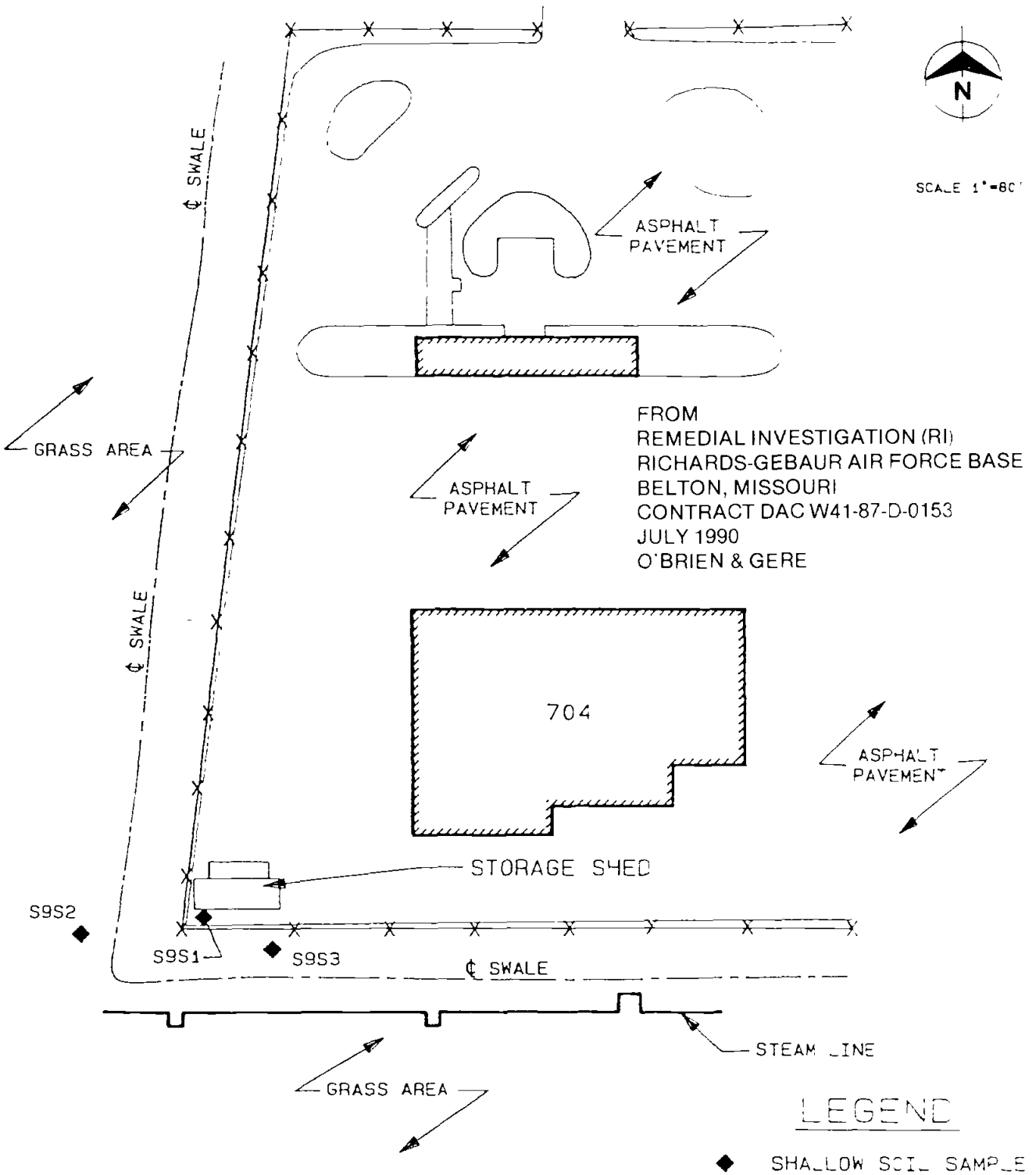

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Figure 4
AUGUST 1989 SAMPLE LOCATIONS. OIL SATURATED AREA SITE SS03

TABLE 1

PREVIOUS ANALYTICAL SAMPLE RESULTS
OIL SATURATED AREA, SITE SS03

DATE	MEDIA	DEPTH	SAMPLE ID	PARAMETER	RESULTS	DETECTION LIMIT
10/86	soil	0'-1'	DF4007	TPH	2000 ppm	1 ppm
				VOCs	ND	1 ppm
				Lead	169 ppm	1 ppm
10/86	soil	0'-1'	DF4008	TPH	3800 ppm	1 ppm
				VOCs	ND	1 ppm
				Lead	117 ppm	1 ppm
10/86	soil	0'-1'	DF4009	TPH	600 ppm	1 ppm
				VOCs	ND	1 ppm
				Lead	343 ppm	1 ppm
10/86	soil	0'-1'	DF4010	TPH	ND	1 ppm
				VOCs	ND	1 ppm
				Lead	14.1 ppm	1 ppm
10/86	soil	0'-1'	DF4011	TPH	2 ppm	1 ppm
				VOCs	ND	1 ppm
				Lead	14.8 ppm	1 ppm
10/86	soil	0'-1'	DF4012	TPH	3 ppm	1 ppm
				VOCs	ND	1 ppm
				Lead	18.5 ppm	1 ppm
10/86	soil	3'-4'	DF4039	TPH	9.0 ppm	1 ppm
				VOCs	ND	1 ppm
				Lead	20.2 ppm	1 ppm
10/86	soil	5'-6'	DF4040	TPH	ND	1 ppm
				VOCs	ND	1 ppm
				Lead	9.22 ppm	1 ppm
10/86	soil	15.5'-16.5	DF4041	TPH	ND	1 ppm
				VOCs	ND	1 ppm
				Lead	10.2 ppm	1 ppm
10/86	water	0'	DF4013	TPH	ND	1 ppm
				VOCs	ND	0.02-2 ppb
				Lead	ND	5 ppb
				TDS	270	NA

TABLE 1

PREVIOUS ANALYTICAL SAMPLE RESULTS
OIL SATURATED AREA, SITE SS03
(con't)

DATE	MEDIA	DEPTH	SAMPLE ID	PARAMETER	RESULTS	DETECTION LIMIT
8/89	soil	0'-1'	S9S1	VOCs	ND	6-12 ppb
				BNAs	ND	0.02-2 ppb
				Lead	94 ppm	.5 ppm
8/89	soil	0'-1'	S9S2	VOCs	ND	6-11 ppb
				BNAs	ND	0 02-2 ppb
				Lead	20 ppm	0 5 ppm
8/89	soil	0'-1'	S9S3/4	VOCs	ND	6-12 ppb
				BNAs	ND	0 02-2 ppb
				Lead	107 ppm	0.5 ppm

estimated to be approximately 20 cubic yards and limited to the top 3 feet (deepest contamination encountered) of soil. Currently, a 6 foot by 16 foot tin shed is located directly over Site SS03.

1.3.2 Hazardous Waste Drum Storage Area - Site SS04

Site SS04 was used for an undetermined number of years for storage of drums of waste oil (primarily waste engine oil) prior to disposal. No hazardous materials have been stored in the area since 1985. A portion of the area was overlain with asphalt in 1989. During the operational period of this waste storage area, soil contamination originated from either transfer spillage, drum overfilling, or drum leaks. Since 1983, Site SS04 has been the subject of three studies which confirmed that Site SS04's contamination is limited to petroleum-based organics (Reference 2).

In October 1986, Site SS04 was tested for purgeable aromatics, purgeable halocarbons, total petroleum hydrocarbons, and lead (see Figure 5) (Reference 2). In August 1989, Site SS04 was tested for purgeable aromatics, purgeable halocarbons, and base neutrals and acid extractables (see Figure 6) (Reference 2). A summary of the analytical results for this Site is compiled in Table 2. The volume of contaminated soil at Site SS04 is estimated to be approximately 23 cubic yards.

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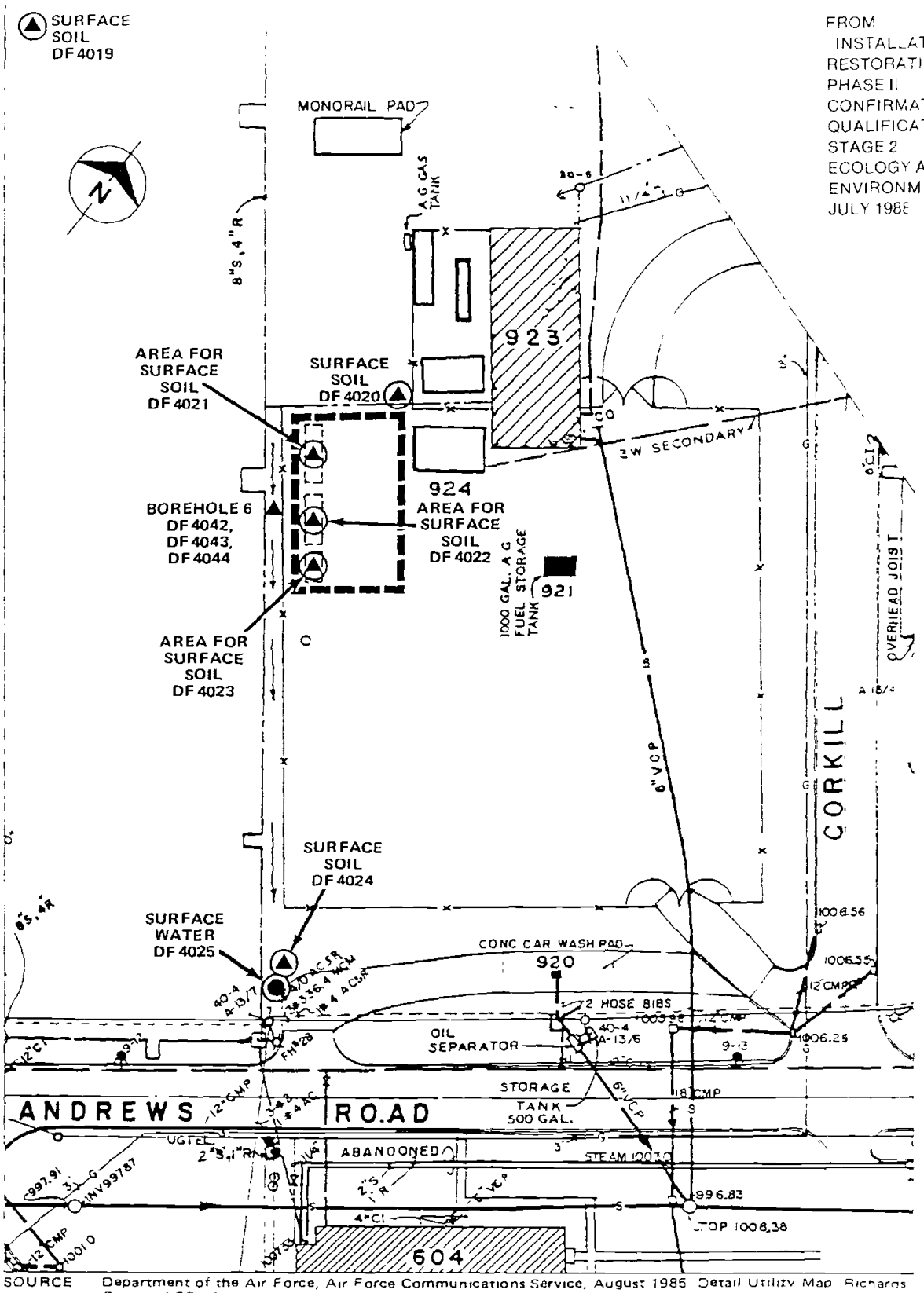

FROM 30 18
INSTALLATION RESTORATION PROGRAM
PHASE II
CONFIRMATION QUALIFICATION
STAGE 2
ECOLOGY AND ENVIRONMENT INC
JULY 1988

SOURCE Department of the Air Force, Air Force Communications Service, August 1985 Detail Utility Map Richards Gebaur AFB, Missouri

KEY.
- Site Boundary
- Surface Runoff Direction
- Subsurface Soil Sampling Location
- Surface Soil Sampling Location
- Groundwater Sampling Location
- Surface Water Sampling Location

SCALE
0 50 100 200 300 FEET

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Figure 5
OCTOBER 1986
SAMPLE LOCATIONS.
HAZARDOUS WASTE
DRUM STORAGE AREA
SITE SS04

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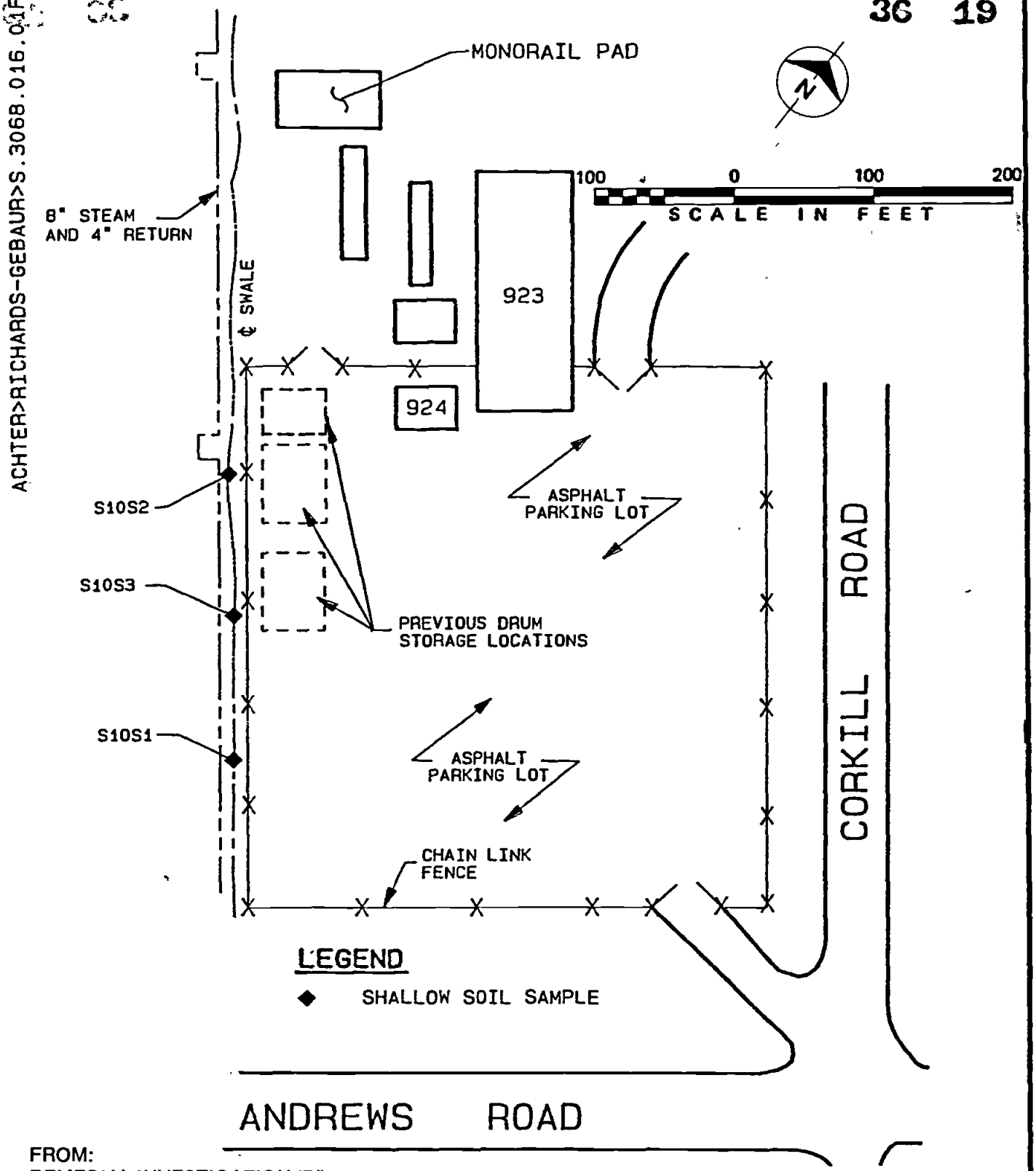

FROM:
REMEDIAL INVESTIGATION (RI)
RICHARDS-GEBAUR AIR FORCE BASE
BELTON, MISSOURI
CONTRACT DAC W41-87-D-0153
JULY 1990
O'BRIEN & GERE

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Figure 6
AUGUST 1989
SAMPLE LOCATIONS
HAZARDOUS WASTE DRUM
STORAGE AREA, SITE SS04

TABLE 2

PREVIOUS ANALYTICAL SAMPLE RESULTS
HAZARDOUS WASTE DRUM STORAGE AREA, SITE SS04

DATE	MEDIA	DEPTH	SAMPLE ID	PARAMETER	RESULTS	DETECTION LIMIT
10/86	soil	0'-1'	DF4019	TPH	ND	1 ppm
				VOCs	ND	1 ppm
				Lead	ND	0.5 ppm
10/86	soil	0'-1'	DF4020	TPH	1900 ppm	1 ppm
				VOCs	ND	1 ppm
				Lead	ND	0.5 ppm
10/86	soil	0'-1'	DF4021	TPH	55 ppm	1 ppm
				VOCs	ND	1 ppm
				Lead	ND	0.5 ppm
10/86	soil	0'-1'	DF4022	TPH	46 ppm	1 ppm
				VOCs	ND	1 ppm
				Lead	ND	0 5 ppm
10/86	soil	0'-1'	DF4023	TPH	140 ppm	1 ppm
				VOCs	ND	1 ppm
				Lead	ND	0.5 ppm
10/86	soil	0'-1'	DF4024	TPH	2.9 ppm	1 ppm
				VOCs	ND	1 ppm
				Lead	0.99	0 5 ppm
10/86	water	0'	DF4025	TPH	ND	1 ppm
				VOCs	ND	2-2 ppb
10/86	soil	0.5'-1.5'	DF4042	TPH	ND	1 ppm
				VOCs	ND	1 ppm
				Lead	0 99	1 ppm
10/86	soil	4.5'-5.5'	DF4044	TPH	1 2 ppm	1 ppm
				VOCs	ND	1 ppm
				Lead	0.99	1 ppm
10/86	soil	9.0'-10.5'	DF4043	TPH	ND	1 ppm
				VOCs	ND	1 ppm
				Lead	0 99	1 ppm

TABLE 2

PREVIOUS ANALYTICAL SAMPLE RESULTS
HAZARDOUS WASTE DRUM STORAGE AREA, SITE SS04
(con't)

DATE	MEDIA	DEPTH	SAMPLE ID	PARAMETER	RESULTS	DETECTION LIMIT
8/89	soil	0'-1'	S10S1	VOCs	ND	6-12 ppb
				BNAs	ND	0.02-2 ppb
				Lead	50 ppm	0.5 ppm
8/89	soil	0'-1'	S10S2	VOCs	ND	6-11 ppb
				BNAs	ND	0.02-2 ppb
				Lead	41 ppm	0.5 ppm
8/89	soil	0'-1'	S10S3/4	VOCs	ND	6-12 ppb
				BNAs	ND	0.02-2 ppb
				Lead	72 ppm	0.5 ppm

2.0 FIELD WORK PLAN

2.0 FIELD WORK PLAN

2.1 GENERAL

Field activities to be conducted at the two sites include pavement removal, the excavation and stockpiling of petroleum and lead contaminated soil, exposed subgrade soil sampling, and the backfilling of the excavations with clean soil. All field activities will be performed at the direction of Burns & McDonnell. The goal for this project is to excavate all soil with a lead content in excess of 238 ppm and a TPH content in excess of 100 ppm

At least ten days prior to the start up of excavation, a digging permit (AF Form 103) will be obtained from the Base Civil Engineering and appropriate utilities will be cleared. Prior to the start of field activities, RGAFB will remove all fencing and structures in the excavation area.

2.2 PAVEMENT REMOVAL

All concrete or asphalt pavement located above the soil to be excavated will be removed prior to the start of excavation. The pavement to be removed is shown in Figures 7 (Site SS03) and 8 (Site SS04). The concrete or asphalt will be demolished and removed in small sections utilizing either a jackhammer or a masonry saw. The demolished concrete or asphalt will be removed and stockpiled separately from the contaminated soil and/or gravel beneath the concrete or asphalt. The removed concrete or asphalt will be stockpiled on plastic sheeting in the immediate area of the excavation.

2.3 SOIL REMOVAL

Upon completion of the pavement removal, the petroleum contaminated soil will be excavated. The soil will be removed to the approximate excavation limits shown on Figures 7 and 8. The soil will be removed in layers and screened with a photoionization detector (PID) to minimize the amount of soil excavated. The soil will be stockpiled on plastic sheeting in the immediate vicinity of the excavation as shown on Figures 7 and 8. The stockpiled contaminated soil will be covered with plastic sheeting (see Figure 9) as protection from weather conditions for 10 days after soil disposition recommendation is provided by Burns & McDonnell to RGAFB. RGAFB will take-on the soil pile caretaker responsibilities after ten days.

2.4 SUBGRADE SAMPLING

Upon completion of the excavations, subgrade sampling will be performed as described in Part 3.0 of this Work Plan. The excavations will remain open while the soil samples are being analyzed for lead and TPH. The excavations will be protected with plastic sheeting to minimize the infiltration of rainwater into the subsurface. Sandbags will be placed around the excavation to isolate the excavation from runoff. Any surface runoff will be pumped out onto the ground surface immediately adjacent to the excavation prior to backfilling.

If laboratory analysis indicates that contaminated soil is still present in the excavation, the contracting officer will determine if further action is needed

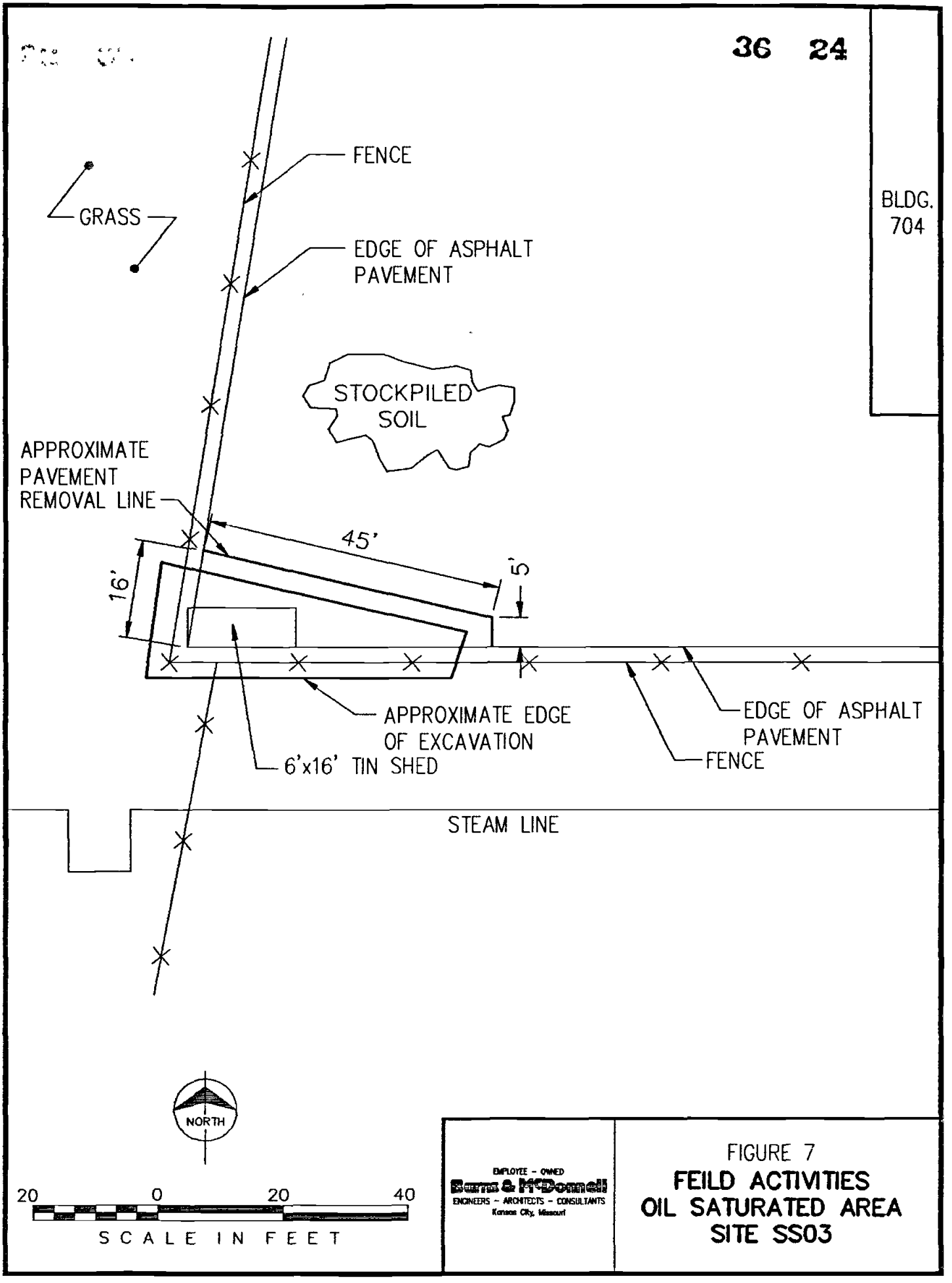
36 24
FENCE
GRASS
EDGE OF ASPHALT PAVEMENT
BLDG. 704
STOCKPILED SOIL
APPROXIMATE PAVEMENT REMOVAL LINE
45'
16'
5'
APPROXIMATE EDGE OF EXCAVATION
6'x16' TIN SHED
EDGE OF ASPHALT PAVEMENT
FENCE
STEAM LINE
NORTH
20 0 20 40
SCALE IN FEET
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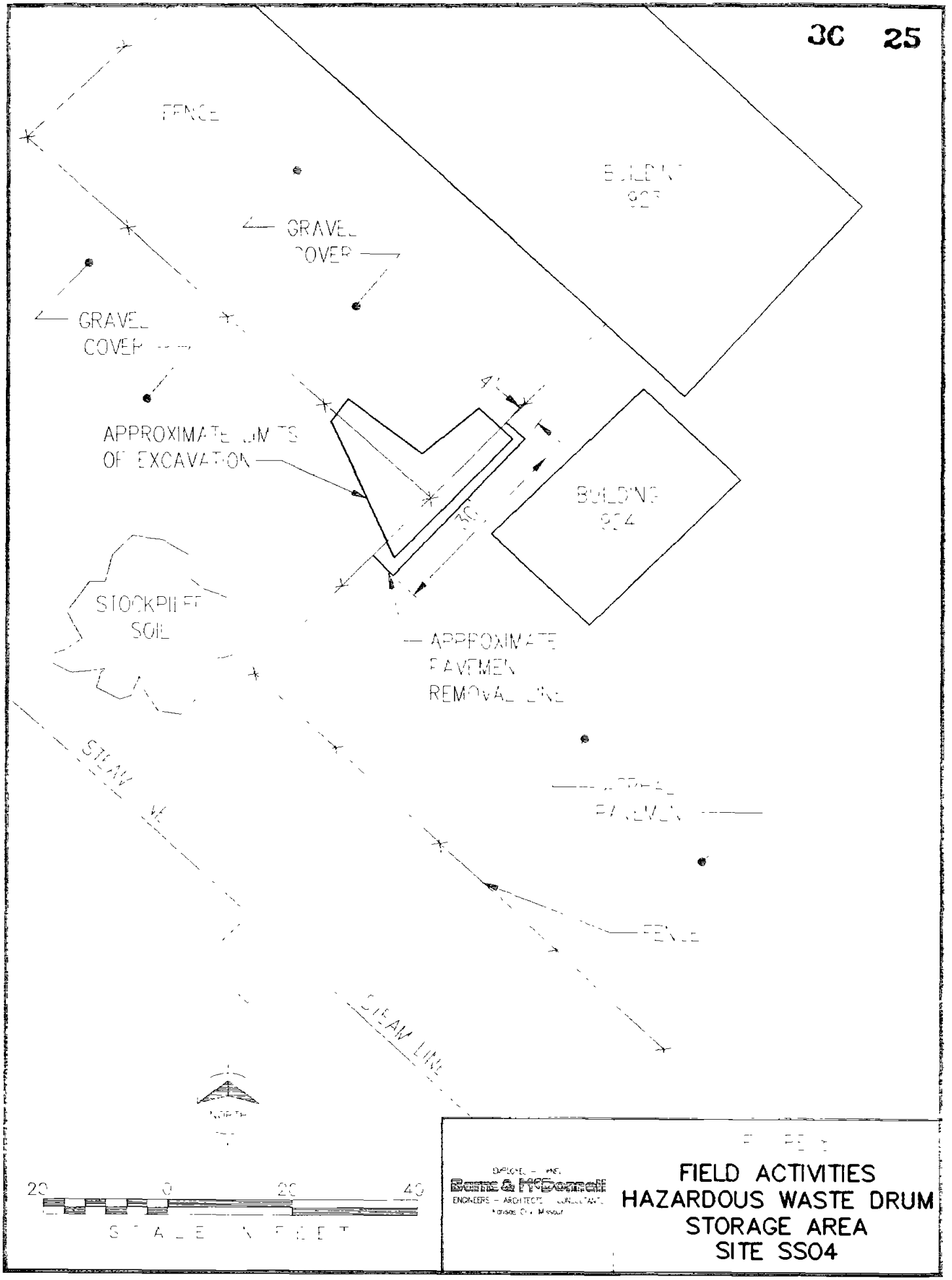
FIGURE 7
FEILD ACTIVITIES
OIL SATURATED AREA
SITE SS03



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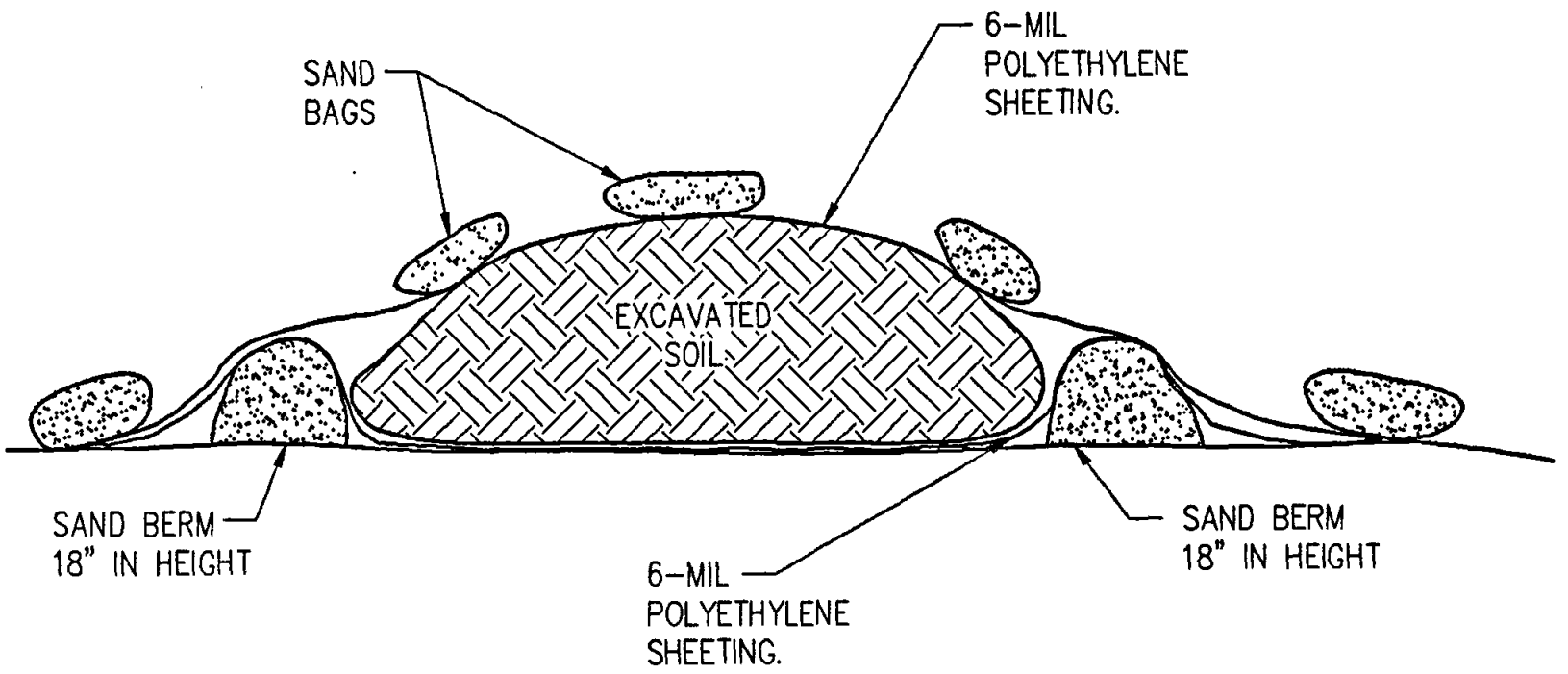
FIELD ACTIVITIES HAZARDOUS WASTE DRUM STORAGE AREA SITE SS04



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Kansas City, Missouri

FIGURE 9
CONSTRUCTION
DIAGRAM FOR
TEMPORARY SOIL PILES

2.5 BACKFILLING THE EXCAVATIONS

The excavations will be backfilled with clean cohesive soil. The soil will be placed in the excavation in loose lifts not exceeding 6 to 8 inches. The soil will be compacted to 95 percent of the maximum standard Proctor density according to ASTM D698. The excavations will be backfilled to the surrounding subgrade elevation.

2.6 RESURFACING OF EXCAVATION AREAS

The excavation areas will be resurfaced to approximately equal the original conditions. At Site SS03, asphalt will be placed in the parking area to the original thickness and the former grassy areas will be sodded. At Site SS04, asphalt and gravel will be placed so that the site is returned to its original condition. No disturbed areas will be left denuded for more than seven calendar days after the extent of contamination is verified by the laboratory analysis results and/or weather conditions permit.

2.7 DECONTAMINATION OF EXCAVATION EQUIPMENT

Equipment used for excavation of contaminated soil will be decontaminated prior to and following excavation of contaminated soil from each area. The decontamination procedures will be conducted at the washrack, Facility 920, shown on Figure 2. The excavation equipment will be decontaminated with a steam cleaner until all visible dirt has been removed.

2.8 PUBLIC AFFAIRS

Neither Burns & McDonnell nor their subcontractors will release to the public any information concerning this project. Any public inquiries made to Burns & McDonnell shall be directed to the RGAFB Contracting Officer. The Contracting Officer will in-turn direct the inquiries to the RGAFB Public Affairs Office.

* * * * *

3.0 SAMPLING AND ANALYSIS PLAN

3.0 SAMPLING AND ANALYSIS PLAN

3.1 SOIL SAMPLE LOCATIONS

Upon completion of the excavations, two grab soil samples will be obtained from representative locations in each excavation. In addition, two composite soil samples will be obtained from the excavated stockpiled soil from each site.

3.2 SOIL SAMPLING PROCEDURES FOR CHEMICAL ANALYSIS

Upon completion of the excavation of the contaminated soil, two soil samples will be collected from each excavation. The sample locations will be chosen based on visual inspection and the field PID readings.

Soil samples will be collected from undisturbed soil in the exposed subgrade of the excavation. Each soil sample will be obtained by placing the soil in the sample container with a precleaned stainless steel spatula. The soil will be packed tightly in the sample container with a minimum amount of airspace present. In addition, for each sample location a sample container will be partially filled and a static headspace reading will be obtained using a properly calibrated PID.

Composite soil samples will be obtained from the excavated soil stockpile for the purpose of determining the proper disposal of the soil. One composite soil sample will be obtained for approximately every 10 cubic yards of excavated soil. The composite soil sample will be composed of approximately 4 equal aliquots. The aliquots will be placed in a precleaned stainless steel bowl and mixed thoroughly. Using a clean spatula, a sample container will be filled from the composited sample. A separate sample container will be partially filled and a static headspace reading will be obtained using a properly calibrated PID

Disposable gloves will be used in handling soil samples to avoid possible cross-contamination of samples. Gloves will be changed between each sample All soil samples obtained will be placed in laboratory cleansed glass sample bottles. The sample bottles will be labeled indicating sample number and sample location. The sample will be stored in an ice chest and chilled to approximately 4°C prior to shipment to the laboratory.

3.3 DUPLICATE AND RINSATE SAMPLES

One duplicate soil sample and one rinsate sample will be obtained and analyzed for TPH and lead. The duplicate soil sample will be collected by placing the soil sample into a clean stainless steel bowl. The sample will be mixed thoroughly and then split into two samples. One sample will be considered the original, while the other is the duplicate. The duplicate sample will be identified with a unique sample identification number and the sample station where the duplicate was collected will be noted in the field logbook

An equipment rinsate blank will be prepared for the sampling equipment used to collect subsurface soil samples for chemical analyses To prepare the

equipment rinsate blank, High-Performance-Liquid-Chromatographic (HPLC) - grade water (ASTM Type II) will be used to rinse the properly decontaminated equipment. The rinsate will be place directly into the container. The equipment rinsate will be analyzed for the same constituents as the primary sample.

3.4 SAMPLE ANALYSIS

All soil and water samples collected will be analyzed for TPH and lead using EPA Methods 418.1 and 7420, respectively.

3.5 SAMPLE PRESERVATION

Soil samples to be analyzed for TPH and lead will be collected in glass jars. The rinsate sample to be analyzed for TPH will be collected in a 16 oz. amber glass jar. The rinsate sample for lead will be collected in an 8 oz. polyethylene container with a HNO_3 preservative. The samples will be stored in an ice chest and chilled to approximately 4°C prior to shipment to the laboratory.

3.6 SOIL SAMPLING EQUIPMENT DECONTAMINATION PROCEDURES

All soil sampling equipment will be decontaminated prior to obtaining each soil sample. The equipment will be cleaned using fresh potable water and Alconox followed by a distilled water rinse.

3.7 SAMPLE CUSTODY AND DOCUMENTATION

Each sample or field measurement will be properly documented to allow timely, correct, and complete analysis. The documentation system provides the means to identify, track, and monitor each sample from the point of collection through final data reporting.

3.7.1 Documentation Procedures

A suitable work area will be established for processing forms and packaging samples. After all sample documentation has been completed, and before the samples are prepared for shipping, a project team member will check the data on all forms and labels, comparing them to the entries in the logbook.

3.7.1.1 Chain-of-Custody Record

The chain-of-custody record is employed as physical evidence of sample custody. The sampler completes a chain-of-custody record to accompany each sample shipment from the field to the laboratory.

The custody record is completed using waterproof ink. Corrections are made by 1) drawing a line thorough the error, 2) initialing and dating the error, and 3) entering the correct information. Erasures are not permissible. A typical chain-of-custody record is shown in Figure 10.

After completion of the chain-of-custody record by the above procedure, the signed original (top) copy of the chain-of-custody recorded is enclosed in a plastic bag (with any other sample documentation) and secured to the inside of the lid. A copy of the chain-of-custody record is retained for the samplers file.

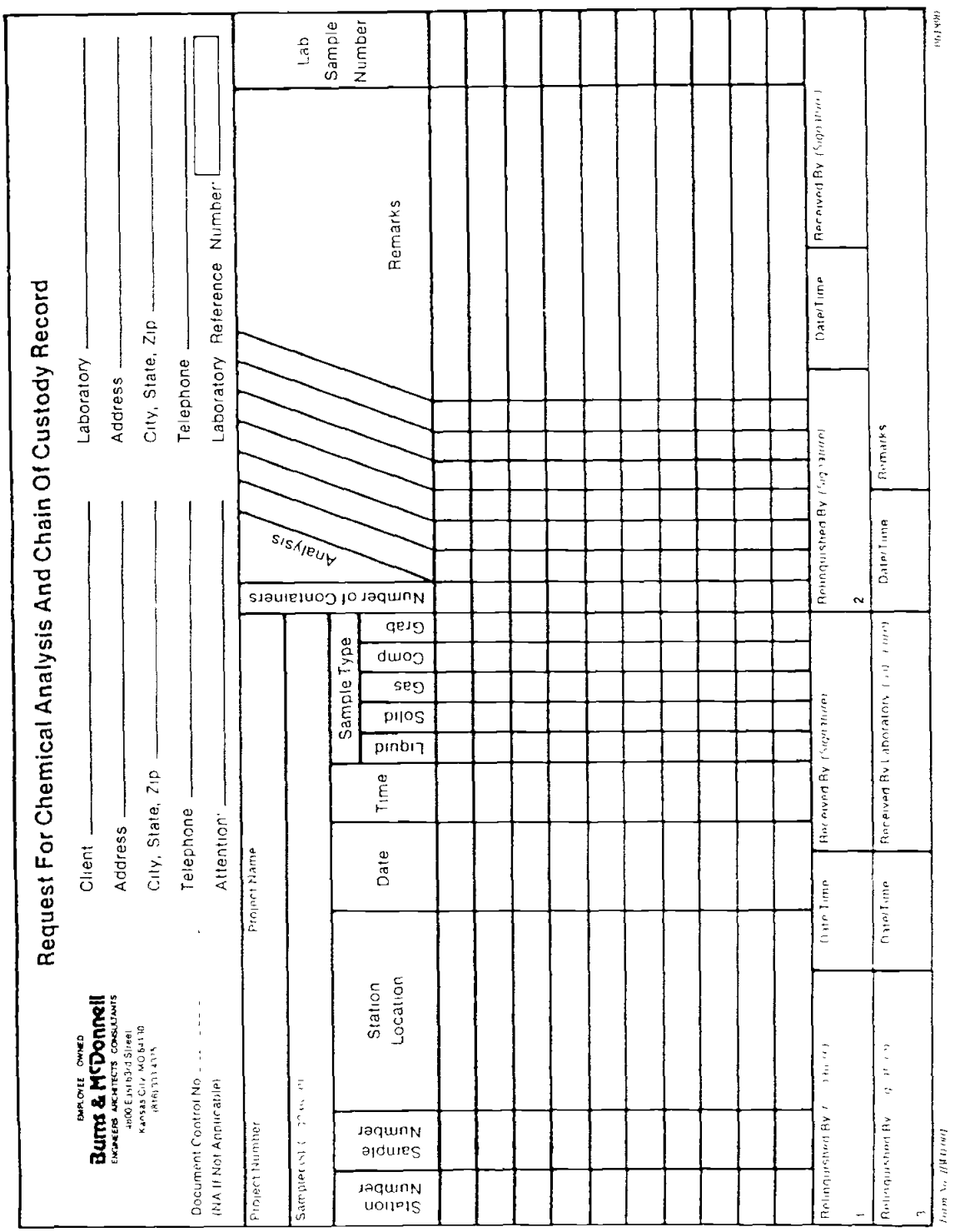


FIGURE 10
CHAIN-OF-CUSTODY RECORD

3.7.1.2 Sample Labels

Each sample removed from a waste site is identified by a sample label containing specific information regarding the sample. Sample labels are retained by the laboratory as physical evidence of sample receipt and analysis. A typical sample label is shown in Figure 11. Each completed sample label will be securely fastened to the sample container.

3.7.1.3 Custody Seals

A custody seal will be used to preserve the integrity of the sample from the time it is collected until it is opened in the laboratory. A typical custody seal is shown in Figure 12.

3.8 CALIBRATION PROCEDURES

To ensure the quality of data collected in the field, all field instruments will be calibrated prior to use. Calibration procedures will follow standard manufacturer's instructions to assure that the equipment is functioning within tolerances established by the manufacturer.

* * * * *

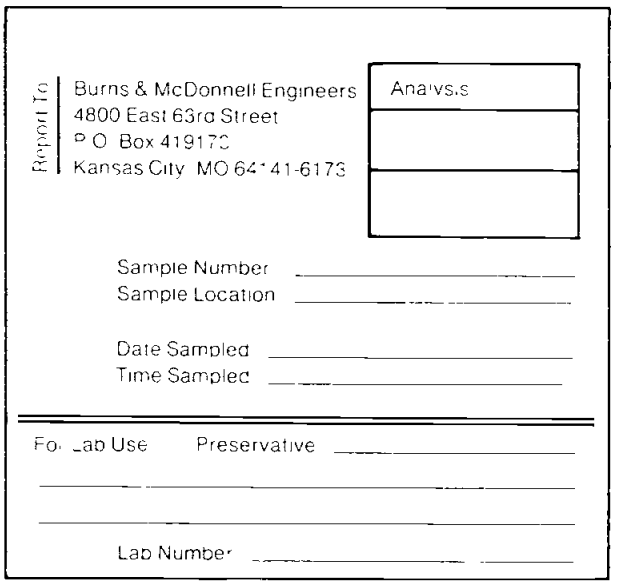


FIGURE 11
SAMPLE LABEL

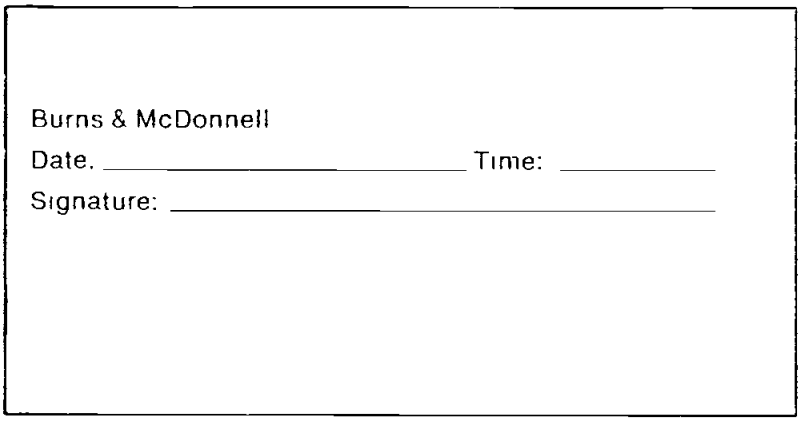


FIGURE 12
CUSTODY SEAL

4.0 QUALITY ASSURANCE PROJECT PLAN

4.0 QUALITY ASSURANCE PROJECT PLAN

4.1 INTRODUCTION

A comprehensive and well-documented Quality Assurance Project Plan (QAPP) is necessary to achieve the appropriate level of precision and accuracy with a minimal expenditure of resources. This QAPP presents objectives and specific QA/QC activities designed to achieve appropriate Data Quality Objectives (DQOs).

To achieve the goals of the QAPP, this Work Plan is based upon CERCLA protocols developed by EPA. The QAPP prepared by the contracted laboratory is found in Appendix A.

4.2 QUALITY ASSURANCE OBJECTIVES

The principal objective of the QAPP is to maintain the quality of operational activities and document the quality of data generated. Experienced and trained on-site personnel will conduct field operations and ship samples to the laboratory. The project will be staffed with personnel experienced in the technical and management disciplines appropriate for the project.

4.2.1 Measurement Objectives

Measurement parameters vary depending upon the circumstances surrounding a specific sampling event, the type and concentration of analytes, and the media to be sampled. All measurements will be conducted to yield consistent results that are representative of the media and conditions measured. Data will be reported in consistent units to facilitate data comparison.

4.2.2 Method Detection Limits

To provide analytical data necessary, the analytical methods employed must be adequately sensitive to detect low levels of contaminants. Method detection limits for each of the analyses proposed are included in the laboratory QAPP provided in Appendix A.

4.2.3 Quality Control Parameters

The quality assurance objective for analytical data is to collect environmental monitoring data of known and acceptable quality. To meet this objective, the QC parameters of precision, accuracy, representativeness, completeness, and comparability will be addressed.

Precision and Accuracy

Precision and accuracy QC limits (in terms of spike recoveries, duplicate results, etc.) must be met for the analytical data to be considered acceptable. These factors are discussed in the laboratory QAPP provided in Appendix A.

Field duplicates will be collected to evaluate the precision of field sampling techniques. The Relative Percent Difference (RPD) between the matrix sample and its duplicate will be compared to the precision limits identified in the laboratory QAPP (see Appendix A).

The control limits specified for accuracy and precision will be utilized to identify outliers (results outside the specified control limits). If any

outliers occur or contamination is detected in the blanks, the corresponding analytical result(s) will be flagged.

The primary objective of field measurement is to obtain reproducible results which have the degree of accuracy consistent with limits imposed by the intended use of the data. Thus, QC procedures for field measurements will be limited to calibration of instruments (where appropriate) and checking the reproducibility of measurements by taking multiple readings.

Representativeness
Representativeness is a qualitative evaluation that assesses whether the information obtained during the investigation accurately represents actual site conditions. The requirement of representativeness was considered during the planning stages and is reflected in the sampling and analysis approach. Representativeness will be assessed after initial data validation and reduction, and will be based only on validated data.

Completeness
Completeness is defined as the percentage of samples collected having valid data. The objective of evaluating completeness is to verify that enough valid data is provided to meet the goals of this remediation. Completeness will be assessed by comparing the number of samples having valid data to the number of samples collected. A completeness of 80 to 85 percent is acceptable for samples other than those designated as "critical" given the project goals.

Comparability
Comparability is assessed to verify that the data developed during the remediation are comparable with applicable hazard criteria and, where appropriate, with data available from other scientific studies in the area.

4.3 SAMPLING PROCEDURES
The common objective of the sampling procedures described in this Work Plan is to obtain samples that represent the environmental matrix to be analyzed. The potential for trace levels of contaminants from external sources to adulterate samples will be minimized through the use of proper sampling techniques and sampling equipment.

Preparation of Sampling Equipment and Containers
All drilling equipment and materials, nondedicated sampling tools, and field measurement instruments will be decontaminated before and after each sampling or field measurement event to prevent sample contamination or faulty field measurements.

All sample containers will be provided by the contract laboratory. Containers provided through this program are precleaned and QC-tested according to prescribed procedures to avoid contamination that could affect sample data results.

Sample Collection, Preservation, Transport, and Storage
Sample collection procedures are discussed in Section 3.0 of this document. Preparation, handling, and shipping of all sample containers will follow CERCLA guidelines as outlined in Section 3.0.

The necessary documentation associated with preparing samples for transport is addressed in Section 3.0. Storage of samples by laboratories will conform to CERCLA procedures

4.4 SAMPLE CUSTODY AND DOCUMENTATION

Each sample or field measurement must be properly documented to allow timely, correct, and complete analysis. These factors are critical to support decisions concerning the site. The documentation system provides the means to identify, track, and monitor each sample from the point of collection through final data reporting. Examples of all relevant field documentation forms are included in Section 3.0.

4.4.1 Laboratory Custody

Laboratory custody will conform to the established procedures identified in the laboratory QAPP provided in Appendix A. These procedures include:

- Designation of a sample custodian.
- Completion (by the custodian) of the Chain-of-Custody Record, sample tag, and laboratory request sheet (including documentation of sample condition upon receipt).
- Laboratory sample tracking and documentation procedures.
- Secure and appropriate storage to maintain sample integrity.
- Proper data logging and documentation, including preservation and custody of all original laboratory records.

4.5 ANALYTICAL PROCEDURES

All samples will be analyzed as specified in Section 3.0. In addition, several field activities will be performed. The ten QAPP elements listed below are pertinent to both laboratory procedures and field activities

QAPP ELEMENTS
Sample Custody
Calibration Procedures
Analytical Procedures
Internal Quality Control
Data Reduction and Validation
Performance and System Audits
Data Assessment
Preventative Maintenance
Accuracy and Precision Definitions
Corrective Action

* * * * *

5.0 HEALTH AND SAFETY PLAN

5.0 SITE HEALTH AND SAFETY PLAN

Project Name: USRGAFB
Identification Number: 91-804-3-008
Location: Richards-Gebaur Air Force Base, Missouri
Date Plan Approved:_____
Reviewer's Name: _____
Reviewer' Signature: _____
Reviewer' Title: _____

5.1 INTRODUCTION

Burns & McDonnell has prepared a Work Plan for the conduct of remediation of the Oil Saturated Area - Site SS03 and the Hazardous Waste Storage Drum Area - Site SS04 on Richards-Gebaur Air Force Base, Missouri. The remediation will consist of soil excavation and the collection of confirmation soil samples. Chemical analysis of soil samples will be conducted.

This Site Health and Safety Plan (SHSP) is intended to cover the field activities for the Work Plan. The health and safety protocols established in this plan are based on the Burns & McDonnell Health and Safety Policy and Procedures for Hazardous Waste Operations, and specific site conditions known and/or anticipated to be present according to available data.

This Site Health and Safety Plan is produced for Burns & McDonnell sampling teams and subcontractor personnel on the project indicated herein. This Health and Safety Plan is specific to the conditions described and the operations to be performed. It might not be appropriate for changed conditions which may be found on different operations (e.g., drilling vs. excavation). Specifications herein are subject to review and revision based on actual conditions encountered in the field during site characterization activities

Before site operations begin, all employees (including subcontractors to Burns & McDonnell) involved in these operations will have read and understood this SHSP and all revisions.

All engineering and subcontractor personnel on-site will have on hand the following documents:

A. 40-hour Hazardous Waste Certificates of Training which adheres 29CFR 1910.120.
B. Certificates of OSHA Eight Hour Refresher Course and Supervisors Course (if applicable).
C. Physician Approval to Wear a Respirator.
D. Current Respirator Fit Test.
E. OSHA Job Poster.

5.2 KEY PERSONNEL AND ALTERNATES

Any changes in personnel will be provided to the Commanding Officer 24 hours prior to the change.

Site Health and Safety Supervisor: Ted Riegel

The Site Health and Safety Supervisor (SHSS) is responsible for maintaining proper medical surveillance, providing hazard communication information, training employees in safe operating procedures, and advising the Project Manager on any matters concerning the health and safety of employees or the public. The SHSS may be required to perform various types of area or personnel monitoring for purposes of verifying worker exposure and proper selection of personal protective equipment. The SHSS should be consulted before any changes in the recommended procedures or levels or protective clothing are made. Usually, changes in the procedures and levels or protection will be made by the SHSS, sometime after consultation with the Health and Safety Department. The form in Appendix B is used for on-site documentation.

Furthermore, the SHSS is to be knowledgeable of the Site Health and Safety Plan. The SHSS will conduct daily health and safety meetings which will review work, hazards, level of protection, and emergency routes. The specific topics discussed in daily safety meetings will be documented on the appropriate Burns & McDonnell form also found in Appendix B.

Project Manager: William Singleton.

The Project Manager has the primary responsibility for the fulfillment of the terms of the contract. The Project Manager will oversee operations and see that safety requirements are met.

Field Site Manager: Lori Wallace

The Field Site Manager is the on-site coordinator and overseer of operations. It is the Field Site Manager's duty to maintain site security, supervise the Burns & McDonnell personnel on site, coordinate the activities of subcontractor personnel, and ensure that all procedures (health and safety, decontamination, protective equipment, etc.) are followed.

The following individuals when located on site will have the authority and responsibility to change levels of protection and when necessary to shut down the operation:

Ted Riegel - Site Health and Safety Supervisor
Lori Wallace - Field Site Manager

5.3 HEALTH RISK ANALYSIS

5.3.1 Job Tasks and Operations

Table 3 is a general summary of the various tasks, operations within each task, potential associated risks and personnel level of protection.

5.3.2 Health Analysis and Chemical Risk Assessment

Table 4 is a summary of the various chemicals which may potentially be encountered, and associated health risks.

Table 3

SUMMARY OF RISKS

Job Task/Operation	Inhalation Hazard	Contact w/Contaminated Soils	Noise Hazard	Heat Stress	Electrical	Potential Fire/Explosion	Contact w/Contaminated Liquids	Cold Stress	Collapsing of Structure on Pers.	Physical Injury	Over Head Power Lines	Buried Tank	Underground Pipe/Utilities	Skin Hazard	Ventilation Problem	Spillage of Liquids/Release	Vandalism	Equipment Freezing Problem	Leaks in Lines	Level of Protection	Air Monitoring
EXCAVATION AND SAMPLING OF SOIL.	X	X	X	X	X	X	X	X	X	X		X	X	X			X	X		D	PHOTOIONIZATION DETECTOR
DECONTAMINATION OF EXCAVATION EQUIPMENT			X	X			X	X		X				X		X		X	X	GLOVES, BOOTS, SARANEX	
PREPARATION OF LABORATORY SAMPLES		X	X						X					X	X	X				GLOVES	

TABLE 4 PAGE#: 1

CHEMICAL HEALTH RISKS

COMMON NAME CAS#	EXPOSURE LIMITS	IDLH/STEL	ROUTE OF ENTRY	SYMPTOMS	PHYSICAL DESCRIPTION	CHEMICAL AND PHYSICAL PROPS	ODOR THRESH (ppm)
LEAD CAS#: 7439-92-1 SYNONYM: LEAD GLOVE MAT: BUTYL, VITON, NITRILE; MANY	0.05mg/M3	VARIABLE	INH., ING,CON	LASSITUDE, INSOMNIA, PALOR, ANOREXIA, COLIC.	VAR., DEP. ON COMPOUND	MW: BP: ND SOL: VAR FLP: ND MP: UEL: NA LEL: NA VP: NA VAP D: IP: NA	NA
PAHs CAS#: NA SYNONYM: POLYNUCLEAR AROMATIC HYDROCARBONS; AS BENZO(a)PYRENE, FLUORANTHENE, CHRYSENE & PYRENE GLOVE MAT: ?butyl	5mg/M Cu	NA - USUALLY CARCINOGENS	Inh (dust), Abs	Photosensitiz; skin cancer, long term; inh-pos lung cancer.	Yellow cryst, tarry subst, as typically enc.	MW: BP: Var SOL: Var FLP: NA MP: VAR UEL: NA LEL: NA VP: INS VAP D: NA IP: NA	NA

KEY: MW-Molecular Weight; FLP-Flash Point, Closed Cup (If Annoted OC, Open Cup)degrees F. BP-Boiling Point ((=low given)
Sol-Solubility in Water UEL-Upper Explosive Limit MP-Melting Point IP-Ionization Potential (* est)
VP-Vapor Pressure LEL-Lower Explosive Limit Vap D-Vapor Density D-Density NA-Not Available

Before excavation, each location will be cleared by Burns & McDonnell personnel utilizing a Field Safety Checklist as found in Appendix B The checklist requires the review of facilities and utility drawings by Burns & McDonnell personnel, utility representatives, and the appropriate digging permit (AF Form 103) from Base Civil Engineering. If each location is clear, then the appropriate people will sign the safety checklist.

5.4 EMPLOYEE TRAINING

5.4.1 General Requirements

All operational employees participate in routine health and safety education and training programs. These programs, directed by the Safety Specialist, are designed to provide these employees with a thorough knowledge of hazardous materials, health and safety hazard potentials and compliance with federal OSHA 29 CFR 1910.120(e)· 40 hours initial instruction, 8 hours annual refresher training, and supervisor's additional 8 hours specialized training. As a minimum, this training includes the following.

- General Safety Rules
- Basics of Chemistry
- Basics of Toxicology/Physiology
- Hazardous Materials (types/characteristics)
- Hazard Communication Information
- Respiratory Protection
- Respirator Training
- Chemical Protective Clothing
- Decontamination Procedures/Personal Hygiene
- Fire Prevention/Protection
- Confined Space Work/Safety
- Atmospheric Testing/Sampling Procedures
- Emergency Response Procedures
- Federal and State Regulations

5.4.2 Site-Specific Training

Burns & McDonnell personnel will provide site-specific training, daily to subcontractor personnel and to the Burns & McDonnell field team. The subcontractor supervisory staff will be responsible for providing basic safety training, including respiratory training, to their own employees. Documentation of subcontractor personnel basic safety training will be provided to Burns & McDonnell prior to excavation beginning. Supervisors will document the daily training, showing subjects discussed and names of all attendees. The subjects will include, but will not be limited to, the following items:

- Protective clothing - correct use, fit, limitation, inspection, repair, and replacement.
- Use of air monitoring equipment.
- Decontamination procedures for personnel.
- Specific action to avoid exposure.
- Special chemical and physical hazards and potential health effects.

The site-specific training will be provided by the SHSS on the first day of field work, prior to commencement of excavating. The SHSS will provide review and update sessions thereafter each day at the beginning of the work day.

5.4.3 Training Documentation

The site-specific training conducted will be documented as to the subjects discussed, names of all attendees and the name of the person conducting the meeting. Form T-10, Safety Training Session Report (see Appendix B), will be completed daily.

Individual training records will be maintained on all personnel involved in the existing and potential hazards of this project. These individual employee safety training records will be maintained current at all times at Burns & McDonnell. Each member of the investigative team will have copies of their training certificates including: 40 hour training, annual refresher, supervisor's training, current fit test record and current physician's approval to wear a respirator.

5.4.4 Site Safety Inspection

The SHSS will assess the effectiveness of the SHSP through periodic evaluation (at least once a day). Changes will be discussed with the Health and Safety Officer (HSO). Any changes to the SHSP will be discussed during the daily review and update sessions.

If field changes are to be made in the SHSP, due to changes in scope or field conditions, then the HSP Field amendment form found in Appendix B will be used. This will require changes to be made by the health and safety department and the signatures of the project manager and an industrial hygienist. Any changes in the SHSP will be reported to the Commanding Officer 24 hours prior to the change.

5.5 PERSONAL PROTECTIVE EQUIPMENT

5.5.1 Air Monitoring

Based on organic vapor detector (PID or FID) readings in the breathing zone, the criteria for levels of protection are as follows:

Reading	Level of Protection
10 ppm above background for more than 2 minutes	C
<10 ppm above background	D or Modified D

The breathing zone is defined as:

- The air directly above the excavation or trench, but within the breathing zone of the employee.
- The air directly surrounding the shovel or digging equipment, but within the breathing zone of the employee.

5.5.2 Levels of Protection

Levels of protection are summarized as follows:

5.5.2.1 Level D

- work uniform
- disposable chemical resistant outer gloves
- chemical resistant boots with steel toe and shank
- disposable outer boot covers
- splash goggles/safety glasses/faceshield* (required during excavation operations)
- hard hat* (required during excavation operations)
- inner disposable latex gloves
- escape mask*

5.5.2.2 Modified Level D

Same as Level D plus disposable chemical resistant clothing (Saranex-Coated Tyvek).

5.5.2.3 Level C

- full-face chemical cartridge respirator (NIOSH approved)
- disposable hooded chemical-resistant clothing
- inner disposable latex gloves
- chemical-resistant boots with steel toe
- disposable boot covers
- hard hat*
- coveralls*
- escape mask*
- two-way radios*
- face shield*

5.5.3 Health and Safety Related Field Equipment On Site

- poly-rope
- small hand tools (pump, pliers, hammer, screwdriver)
- photoionization detector with calibration standard
- exclusion zone tape or cones
- lanolin hand cleaner and paper towels

*Optional Equipment

5.6 HEALTH SURVEILLANCE PROGRAM

5.6.1 Employee Medical Examinations

All employees involved with this project work will participate in a medical surveillance program under the direction of an Occupational Physician. This program will include an annual medical evaluation. The annual evaluation will consist of the following:

- Comprehensive Health and Exposure History
- Physical Evaluation
- Urinalysis
- SMAC 24 including total cholesterol and High Density Lipoproteins and GGTP
- Complete blood count (CBC), differential hematocrit, and hemoglobin
- Chest X-ray (every third year)
- Pulmonary Function Testing
- Audiometry
- Vision Testing (distant, near, color)
- Electrocardiogram

Additionally, each employee will be evaluated to determine if physically able to perform work while using respiratory protective equipment in compliance with 29 CFR Part 1910.134 and ANSI Z88.2 - 1980.

A post project follow-up exam may be required if there is a reported exposure incident. This will be determined by the SHSS and the manager of the health and safety group.

5.6.2 Heat Stress

The following information describes the recommended procedures for heat stress monitoring of Burns & McDonnell personnel engaged in field work activities. This procedure applies to all employees who perform field work during hot weather or in hot environments and who may be at risk of developing heat stress. All Burns & McDonnell field personnel will participate in First Aid training in Heat Stress recognition and treatment (American Red Cross - Standard First Aid).

5.6.2.1 Method - Monitoring and Control

Heat stress is the physiological response observed when the body becomes overheated contributing to any of the following conditions; heat strain, heat cramps, heat exhaustion, and heat stroke. A number of signs and symptoms can result such as fatigue, clumsy movement, anxiety, decreased concentration, poor dexterity, irritability, elevated core temperature and unconsciousness. Because heat stress depends on a number of factors such as age, fitness, pre-existing medical conditions, and acclimatization; all workers, even those not wearing impermeable clothing, should be monitored.

Procedure 1

The employee's pulse rate will be used to monitor their individual response to environmental or internal heat load. To measure the heart rate (pulse), have the individual employee monitor themselves (procedure below). This monitoring program will become effective when the ambient work area temperature exceeds 70 degrees Fahrenheit. The pulse rate will be monitored at the beginning and end of each shift and during each rest break.

1. Heart Rate: The wrist pulse is counted for 60 seconds prior to the beginning of the shift and as early as possible during each rest period. (Do not use thumb to monitor another person's pulse rate.)

2. After the first 60 minutes of work, the pulse rate will again be measured to determine the rest break time, duration, and frequency. If the pulse rate is 110 beats per minute or greater, immediately begin the first rest break. Each rest break should be 10-15 minutes (recommended) in duration or until the resting pulse rate falls below 110 beats per minute.

3. Follow these guidelines to determine work-rest period durations. The pulse rate measured at the beginning of the rest break is compared with the chart to determine the work duration before another break is required. For example, if after the first 60 minutes of continuous work the pulse rate is

126 beats per minute, the employees should rest and drink fluids until the pulse rate is below 110 beats per minute (estimated time - 10-15 minutes). Compare the initial rate of 126 to the chart. The chart indicates that work can begin and continue for 1 hour before another rest break is necessary. At the beginning of the next break, the pulse rate is again measured for use in determining the next work-rest schedule. This procedure should be used throughout the work shift.

Heart Rate	90-100	100-110	110-120	120-130	130-140	140-150	Above 150-180
Work Time > 8 hrs (continuum)	4 hrs	2 hrs	1 hour	30 min	15 min	4.6 min	

Pulse Rates 60-90 beats per minute are considered normal and regularly scheduled work hours are recommended.

For unacclimatized workers, the lower pulse rate from each range should be used for the first two weeks.

5.6.2.2 **Prevention of Heat Stress**

Proper training and preventative measures will help avert serious illness and loss of productivity. Preventing heat stress is particularly important because once an individual suffers from heat stroke or heat exhaustion, that person may be predisposed to additional heat related injuries. One or more of the following recommendations will help reduce the potential for heat stress:

1. Adjust Work Schedules:
   a. Modify work-rest schedules according to monitoring.
   b. Mandate work slowdowns as needed.
   c. Rotate Personnel: Alternate job functions to minimize overstress or overexertion at one task.
   d. Add additional personnel to work teams.
   e. Perform work during cooler hours of the day if possible or at night if adequate lighting can be provided.

2. Provide shelter (air-conditioned, if possible) or shaded areas to protect personnel during rest periods. The rest area must be cooler than the work area.

3. Maintain worker's body fluids at normal levels. This is necessary to maintain proper cardiovascular functioning. Daily fluid intake must approximately equal the amount of water lost as sweat, (i.e., drink 8 fluid ounces of water for approximately every 8 ounces of weight lost). The normal thirst mechanism is not sensitive enough to stimulate our desire to drink replacement fluids. When heavy sweating occurs, encourage the worker to drink more. The following tips will be helpful:
   a. Maintain drinking water temperature at 50° to 60°F.
   b. Provide small disposable cups that hold about 4 ounces or their own disposable bottle. Have workers drink 16 ounces of fluid (preferably water, dilute drinks or electrolyte

balanced drinks) before beginning work.

c. Urge workers to drink a cup or two every 15 to 20 minutes, or at each break. A total of 1 to 1.6 gallons of fluid per day are recommended, but more may be necessary to maintain body weight.

d. (Alternate Method) Weigh workers before and after work to determine if fluid replacement is adequate.

4. Encourage workers to maintain level of physical fitness:

a. Acclimatize workers to site working conditions; temperature, wind direction, protective clothing, and workload.

b. Urge workers to maintain normal weight levels.

c. No alcoholic beverages. This should be stressed for off-duty hours.

d. Personnel may want to wear cotton long underwear under chemical protective clothing. Cotton will aid in absorbing perspiration and will hold it close to the skin, which will provide the maximum amount of cooling from the limited evaporation that takes place underneath the chemical resistant clothing.

6. Provide cooling devices to aid natural body heat exchange during prolonged work or severe heat exposure. Cooling devices include:

a. Field shower or hose-down areas to reduce body temperature and/or to cool off protective clothing.

b. Cooling jackets, vests, or suits.

7. Train workers to recognize and treat heat stress. As part of training. identify the signs and symptoms of different heat stress conditions. During times of heat stress conditions, ice should be readily available to rapidly cool victims.

a. Heat Rash
Caused by continuous exposure to heat and humid air and aggravated by chafing clothes. Symptoms include a decreased ability to tolerate heat as well as a rash.

b. Heat Cramps
Caused by profuse perspiration with inadequate fluid intake and chemical replacement (electrolytes). Signs: muscle spasm and pain in the extremities and abdomen.

c. Heat Exhaustion
Caused by increased stress on various organs to meet increased demands to cool the body. Signs: shallow breathing; pale; cool moist skin; profuse sweating; dizziness and lassitude (sluggish or lethargic); nausea and fainting.

d. Heat Stroke
The most severe form of heat stress. Temperature regulation fails and the body temperature rises to critical levels (104°F to 108°F). The body must be cooled immediately to prevent severe injury or death. Medical help must be obtained immediately at a hospital via ambulance. Signs and symptoms are: red, hot, dry skin; no perspiration; nausea;

dizziness and confusion; strong, rapid pulse; and coma
Have ice chests with ice on hand with towels to cool down the body of a heat stroke victim.

5.6.3 Cold Stress Monitoring

The threshold limit values (TLV) guidelines established by ACGIH will be used to prevent cold stress (hypothermia) and cold injury to body extremities (hands, feet and head). The TLV objective is to prevent body core temperature (rectal) from falling below 36°C (96.8°F).

5.6.3.1 **Training**

Appropriate response personnel (investigation, remedial action and sampling) will receive training developed by the American Red Cross in First Aid and cardiopulmonary resuscitation (CPR). These training courses are certified for three years and one year, respectively.

5.6.3.2 **Environmental Monitoring**

The Site Health and Safety Supervisor shall monitor environmental conditions using guidelines established by the ACGIH.

5.6.3.3 **Warm-Up Breaks**

The ACGIH recommended work/warm-up schedule for properly clothed workers at temperatures below freezing shall be followed.

5.6.3.4 **Protective Clothing**

Exposed skin surfaces must be protected by the use of appropriate cold weather protective clothing. These protective items can include facemask, handwear and footwear. Windbreaks can shield the work area from the cooling effects of wind. The workers shall wear protective clothing appropriate for the level of cold and planned physical activity. The objective is to protect all parts of the body with emphasis on hands and feet. Eye protection against glare and ultraviolet light will be worn in snow and/or ice terrain.

5.6.3.5 **Identification and Treatment of Cold Stress**

- Symptoms of Frostbite
  Frostbite usually begins with numbness and/or pain in the extremities or exposed skin surfaces and a decrease in manual dexterity.

- Symptoms of Cold Stress (Hypothermia)
  In hypothermia, the body core temperature is reduced. Hypothermia will very likely result in reduced mental alertness, reduction in rational decision making, fatigue drowsiness and loss of consciousness with a threat of fatal consequences. During exposure to cold, severe shivering develops as a symptom of cold stress.

- First Aid
  The onset of heavy shivering, frostbite, excessive fatigue or other symptoms of cold stress are indications for immediate return to the on-site trailer. The outer layer of clothing should be removed and the remainder of the clothing loosened to

permit sweat evaporation. Warm, sweet drinks and soups should be consumed to provide caloric intake and fluid volume. Oral temperature will be determined, and the worker will be transported to a medical facility if the oral body temperature is below 35.6°C (96°F).

5.7 AIR MONITORING PROTOCOL

5.7.1 Protocols

The following is the protocol to be followed for air sampling at the site.

Organic Vapor Detector

Burns & McDonnell will conduct air monitoring during excavation operations with an organic vapor detector, calibrated for benzene. The instrument will be zeroed to background conditions prior to use.

If the organic vapor detector measures over 10 ppm (parts per million) organic vapors in the working areas for over 2 minutes, the SHSS will halt operations, and respirators and protective clothing will be donned. Respirators can be removed when the SHSS determines that the organic vapor concentrations are consistently close to background.

5.7.2 Instrumentation and Calibration

Air monitoring for the site will be accomplished with the following:

	Calibration Check	
Type	Frequency	Gas Standard
Organic Vapor Detector (PID)	Each day	Isobutylene (100 ppm) (Span adjusted to benzene)

Air Monitoring Frequency

The monitoring equipment will be operated on the following frequencies for these locations:

- Organic vapor detector - periodic, during excavation activities.

5.8 SITE SECURITY AND CONTROL

Restricted site areas shall include, but not necessarily be limited to, the following:

- Zone of exclusion.
- Contamination reduction zone.
- Support zone.

The Zone of Exclusion and Contamination Reduction Zone will be within a 20-foot radius of the excavation operation and will be demarcated by tape. The SHSS will restrict access to this area to site investigation personnel. The personnel documentation station will be located at the entrance to the demarcated area.

The support zone includes the areas surrounding the zone of exclusion and contamination reduction zone.

5.9 DECONTAMINATION PROCEDURES

5.9.1 Personnel Decontamination

5.9.1.1 Level D

- Segregated equipment drop
- Remove disposable outer boot covers
- Remove chemical resistant outer gloves
- Remove hard hat, goggles/safety glasses/faceshield
- Remove inner disposable gloves

5.9.1.2 Level C (and Modified Level D)

- Segregated equipment drop
- Remove disposable outer boot covers
- Remove chemical resistant outer gloves
- Remove chemical resistant suit
- Remove first pair of disposable latex gloves
- Remove respirator/hard hat/faceshield
- Remove second pair of disposable gloves

All disposable contaminated clothing will be collected and disposed of properly. Respirators will be cleaned and disinfected after each day's use or more often, if necessary.

5.9.2 Minimum Equipment Requirements

- Plastic sheeting
- Wash tubs
- Water source

5.10 STANDARD OPERATING PROCEDURES

5.10.1 Personnel Precautions

1. Eating, drinking, chewing gum or tobacco, smoking, or any practice that increase the probability of hand-to-mouth transfer and ingestion of material is prohibited in any area designated contaminated.
2. Hands and face must be thoroughly washed upon leaving the work area
3. Whenever decontamination procedures for outer garments are in effect, the entire body should be thoroughly washed as soon as possible after the protective garment is removed.
4. No facial hair which interferes with a satisfactory fit of the mask-to-face seal is allowed on personnel required to wear respirators.
5. Contact with contaminated or suspected contaminated surfaces should be avoided. Whenever possible, do not walk through puddles, leachate, discolored surfaces, kneel on ground, lean, sit, or place equipment on drums, containers, or the ground.
6. Medicine and alcohol can potentiate the effects from exposure to toxic chemicals. Prescribed drugs should not be taken by personnel at hazardous waste operations where the potential for absorption, inhalation, or ingestion of toxic substances exists unless specifically approved by a qualified physician Alcoholic beverage intake should be minimized or avoided.

7. All personnel must be familiar with standard operating safety procedures and any additional instructions and information contained in this SHSP.
8. Contact lenses cannot be worn when respirator protection is required or when the hazard of a splash exists.
9. Personnel will be aware of symptoms for toxic chemicals on site and for heat or cold stress.
10. Respirators shall be cleaned and disinfected after each day's use or more often if necessary.
11. Prior to donning, respirators will be inspected for worn or deteriorated parts. Emergency respirators on self-contained devices will be inspected at least once a month and after each use.
12. The employee will be familiar with all sections of the established respirator program.

5.10.2 Operations

1. All personnel going on site must be adequately trained and thoroughly briefed on anticipated hazards, equipment to be worn, safety practices to be followed, emergency procedures and communications.
2. Any required respiratory protective devices and clothing must be worn by all personnel going into areas designated for wearing protective equipment.
3. Personnel on site must use the buddy system when wearing respiratory protective equipment. As a minimum, a third person, suitably equipped as a safety backup, is required during extremely hazardous entries.
4. Visual contact must be maintained between pairs on site and safety personnel. Entry team members should remain close together to assist each other during emergencies.
5. During continual operations, on-site workers act as safety backup to each other. Off-site personnel provide emergency assistance.
6. Personnel should practice unfamiliar operations prior to doing the actual procedure.
7. Entrance and exit locations must be designated and emergency escape routes delineated. Warning signals for site evacuation must be established.
8. Communications using radios, hand signals, or other means must be maintained between initial entry members at all times. Emergency communications should be prearranged in case of radio failure,necessity for evacuation of site, or other reasons.
9. Wind indicators visible to all personnel should be strategically located throughout the site.
10. Personnel and equipment in the contaminated area should be minimized, consistent with effective site operations.
11. Work areas for various operational activities must be established.
12. Procedures for leaving a contaminated area must be planned and implemented prior to going on site. Work areas and decontamination procedures must be established based on expected site conditions.

13. Frequent and regular inspections of site operations will be conducted to insure compliance with this SHSP If any changes in operation occur, the SHSP must be modified to reflect changes
14. All electrical equipment (power tools, extension cords, instruments, radios, etc.) shall conform to the section anticipated for OSHA 29 CFR Part 1926.400 Subpart K.
15. Fire prevention and protection (appropriate signs for flammable liquids, smoking areas, storage areas of combustible or flammable materials, etc.) shall be in accordance with OSHA 29 CFR Part 1926.150 Subpart F.

5.10.3 Excavation Operations

1. All personnel on site during excavation operations must be adequately trained and familiar with the hazards associated with trenching and excavation work.
2. All operations will be performed in accordance with the Occupational Safety and Health Administration's standard on Excavations, 29 CFR 1926.650.
3. All utilities must be identified and located prior to opening the site
4. Adequate means of access and egress shall be maintained for personnel and equipment from the excavation site. Trenching operations that are 4 feet or more in depth must have a safe means of egress and access to limit lateral travel to less than 25 feet.
5. Warning signals for site evacuation must be established.
6. Where public vehicular traffic posses a threat, all employees will be provided with reflective or high visibility warning vests.
7. Personnel must obey all existing barriers, restricted entrance areas, and warning signs (i.e., elevated loads, hazardous atmospheres, water accumulation, loose rock or soil)
8. Emergency rescue equipment must be readily available and on site
9. A competent person (as defined in 29 CFR 1926.650) shall make daily inspections of the excavation site prior to each day's work and as necessary throughout the shift
10. Fall protection shall be provided at the edge of all sites and on permitted walkways crossing the site.
11. Where necessary, a protective system will be designed and implemented to prevent cave-ins. The system will be dependent on depth, soil conditions and use of the following; sloping and benching, timber or aluminum hydraulic shoring
12. All mobile equipment (in addition to digging equipment) used on site must be equipped with audible back-up alarms

5.11 CONTINGENCY PLAN

Copies of the following will be posted in site trailers, kept in all site vehicles, and provided to personnel in charge on site.

5.11.1 Emergency Action - Standard Operating Procedures

1. Name, address, and telephone number of the nearest medical treatment facility should be conspicuously posted. A map and directions for locating the facility, plus the travel time. should be readily available.

2. Arrangements to quickly obtain ambulance, emergency, fire, and police services. Telephone numbers and procedures for obtaining these services should be conspicuously posted.
3. Prior to mobilization at the site, personal contact should be made with emergency room personnel, the Poison Control Center, the local fire department and police. If outside of an established town, contact shall be made with county officials and local emergency services.
4. Emergency showers, eye wash fountains, and first aid equipment will be readily available on site. Personnel should have first aid and medical emergency training.
5. Provisions must be made for the rapid identification of the substance to which the worker has been exposed. This information must be given to medical personnel.
6. Procedures must be made for decontamination of injured workers and preventing contamination of medical personnel, equipment, and facilities.
7. Sufficient water and/or dry chemical fire extinguishers and neutralizing agents will be maintained on site to cope with any situation until emergency services can arrive.

5.11.2 Medical Emergencies

1. Any person who becomes ill or injured in the exclusion zone must be decontaminated as well as possible and giving consideration which risk will be greater, the spread of contamination or the health of the individual. If the injury or illness is minor, full decontamination should be completed and first aid administered prior to transport. If the patient's condition is serious, at least partial decontamination should be completed (i.e., complete disrobing of the victim and redressing in clean coveralls or wrapping in a blanket). First aid should be administered while awaiting an ambulance or paramedics. The SHSS and the Field Site Manager are trained and certified in First Aid and CPR and will be on site.
2. Anyone being transported to a clinic or hospital for treatment should take with them information on the chemical(s) to which they have been exposed at the site.
3. Any vehicle used to transport contaminated personnel, will be tested and cleaned as necessary by an investigation team member appointed by the Field Site Manager, if so requested.

5.11.3 First Aid Measures

In the event that personnel exposure symptoms occur, the following procedures will be used:

Petroleum Products

Eye Contact: Flush eye immediately with copious amount of water; repeat until irritation is eliminated. If prolonged irritation occurs for more than 15 minutes, seek medical attention.

Skin Contact: Wash exposed area with soap and water. If dermatitis or severe reddening occurs, seek medical attention.

Inhalation: Remove person into fresh air. If symptom occurs for more than 15 minutes, seek medical attention.

Ingestion: Do not induce vomiting, seek immediate medical attention.

IN CASE OF SERIOUS INJURY OR ILLNESS: In case of any injuries or illness, call 9-911.

DIRECTIONS TO NEAREST HOSPITAL: See attached Figure 13.

NOTE: Emergency routes to be verified and driven prior to any site activities.

5.11.4 Flammable Conditions

In the event that combustible vapors exceed 20 percent of the lower explosion limit (LEL), or strong odors are detected in the excavation site, the following actions should be taken:

- Eliminate all ignition source, no smoking, cutoff electric switches away from odors. Do not turn on/off electric switches if strong odors are present, unless the switch is intrinsically safe. Do not allow cars to operate or travel over manholes.
- Remove personnel away from the excavation site
  Call: In the listed sequence
  - Base Environmental Coordinator - Kent Kerr: 348-2442
  - Project Engineer - J. D. Langford: 822-3175
- Allow excavation site to ventilate.
- Approach excavation cautiously with combustion gas indicator turned on and take continuous readings
- If readings are greater than 20%, make provisions to ventilate with an intrinsically safe fan or copus airject eductor-type fan (Pneumatic driven).
- Provide answering personnel with the call back number(s). locations, directions, and situation assessment.

5.11.5 Chemical Emergencies

The following conditions will necessitate the cessation of field work in the area of concern and revisions to this SHSP.

- Observation of large quantities of unidentified waste
- Readings of over 500 ppm on the organic vapor detector (in the breathing space).

5.11.6 List of Emergency Response Contacts

RGAFB IN-HOUSE EMERGENCY SERVICES
(on non-house phones dial 348 and extension)

FIRE	ext	2117
SECURITY (POLICE)	ext	2118
DISASTER PREPAREDNESS	ext	2008/2444
BASE OPERATION CENTER	ext	2010

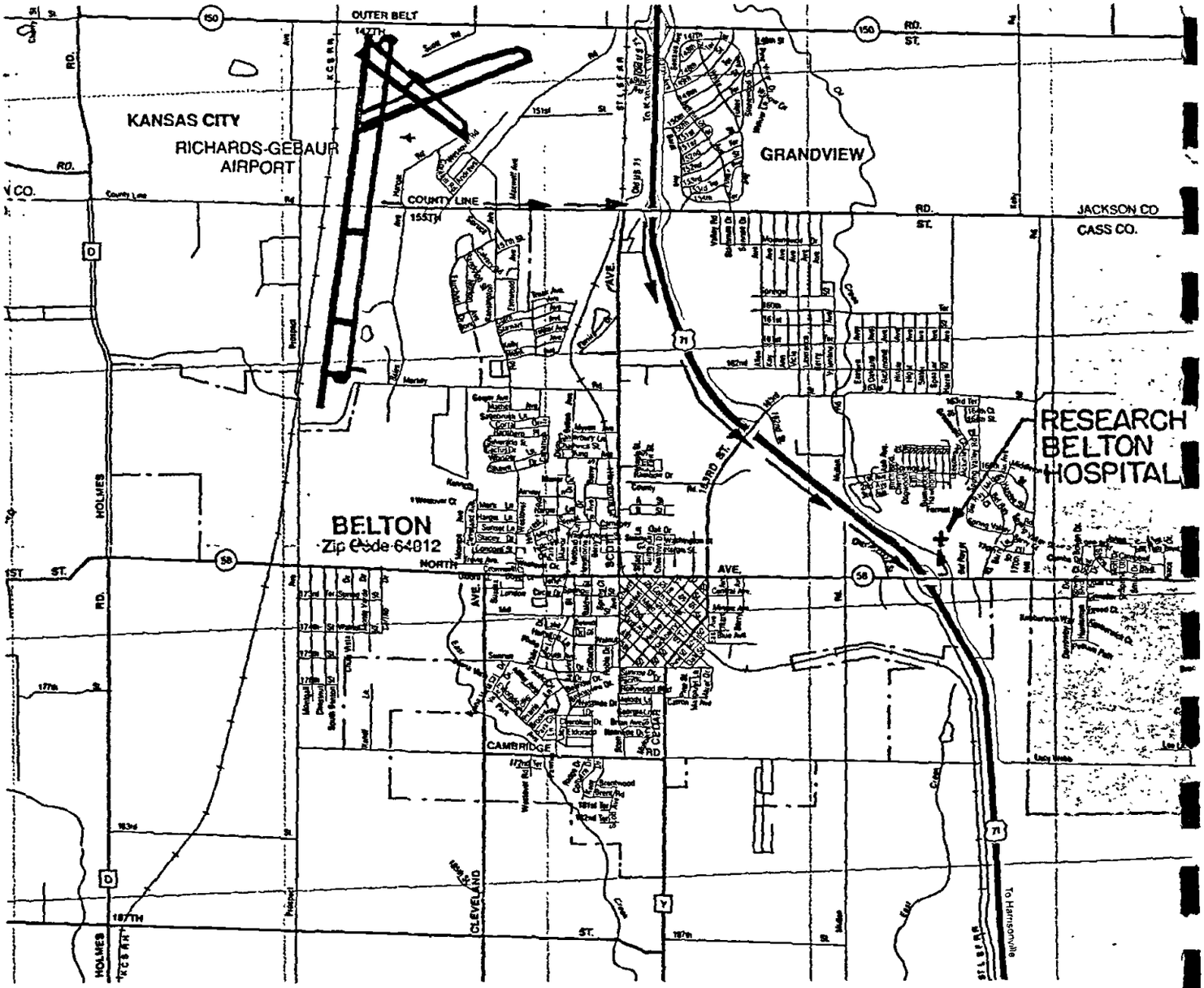


FIGURE 13
LOCATION AND ROUTE TO AREA HOSPITAL

BURNS & McDONNELL EMERGENCY NUMBERS
(on house phones, dial 9 and number)

FIELD OBSERVER: Lori Wallace	333-4375
SITE SAFETY OFFICER: Ted Riegel	333-4375
HEALTH AND SAFETY GROUP MANAGER: Mary Murphy	822-3228
PROGRAM MANAGER: William Singleton	822-3133
PROJECT ENGINEER: J. D. Langford	822-3175
VICE PRESIDENT WASTE MANAGEMENT DIVISION: John Ruf	822-3106

Any change in Burns & McDonnell Emergency Numbers will be reported to the Commanding Officer 24 hours prior to change.

LOCAL EMERGENCY SERVICES
(on house phones, dial 9 and number)

KANSAS CITY FIRE DEPARTMENT	911
AMBULANCE SERVICE	911
KANSAS CITY POLICE DEPARTMENT	911
MISSOURI HIGHWAY PATROL	911 or 524-9200
ST. JOSEPH HEALTH CENTER (HOSPITAL)	911 or 942-4400

* * * * *

REFERENCES

1. "Installation Restoration Program Records Search for Richards-Gebaur Air Force Base", CH2M Hill, March 1983.

2. "Remedial Investigation, Richards-Gebaur Air Force Base, Belton, Missouri Contract DACW41-87-D-D153", O'Brien & Gere, July 1990

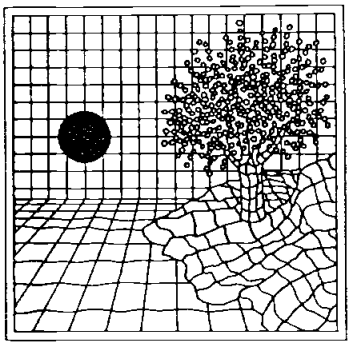
3. USEPA, 1987, Data Quality Objectives for Remedial Response Activities Development Process, U.S. EPA, Washington, D.C., March 1987 (EPA/540/G-87/003).

4. USEPA, 1988, Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA, Office of Emergency and Remedial Response, Interim Final (EPA/540/G-89/004)

* * * * *

APPENDIX A

Laboratory Standard Operating Procedures



SOUTHWEST LABORATORY OF OKLAHOMA, INC.

QUALITY ASSURANCE PROJECT PLAN

Prepared for

BURNS & McDONNELL

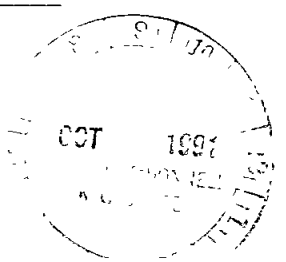
USRGAFB

October 9, 1991

APPROVAL:

QA/QC Officer _____ Date 10-9-91

1700 WEST ALBANY, SUITE C • BROKEN ARROW, OK 74012
(918) 251-2858 • FAX (918) 251-2599



CONTENTS

Copies of this document are in the possession of Southwest Laboratory of Oklahoma's Quality Assurance Officer, Chuck Hoover, Southwest Laboratory of Oklahoma's Administrative Office, and Burns & McDonnell.

TABLES & FIGURES

INTRODUCTION

SOUTHWEST LABORATORY OF OKLAHOMA (SWLO) is pleased to present a Quality Assurance Project Plan for Burns & McDonnell for the USRGAFB Project.

Founded in 1976, Southwest Laboratory has two local offices and four affiliated locations to provide a complete range of petroleum and environmental analysis. SWLO has extensive experience in the analysis of water, wastewater and soil samples under RCRA and CERCLA regulatory requirements. Our personnel are highly trained and experienced professionals capable, not only performing the analysis, but assuring the quality of the data.

For the referenced project SWLO will utilize our Southwest Laboratory of Oklahoma, Broken Arrow facility. The SWLO Broken Arrow facility occupies over 32,000 square feet of laboratory and office space. This facility presently employs over eighty-five people. The TPH and volatile organics will be conducted at our Tulsa facility. All other work will be conducted at the Broken Arrow facility.

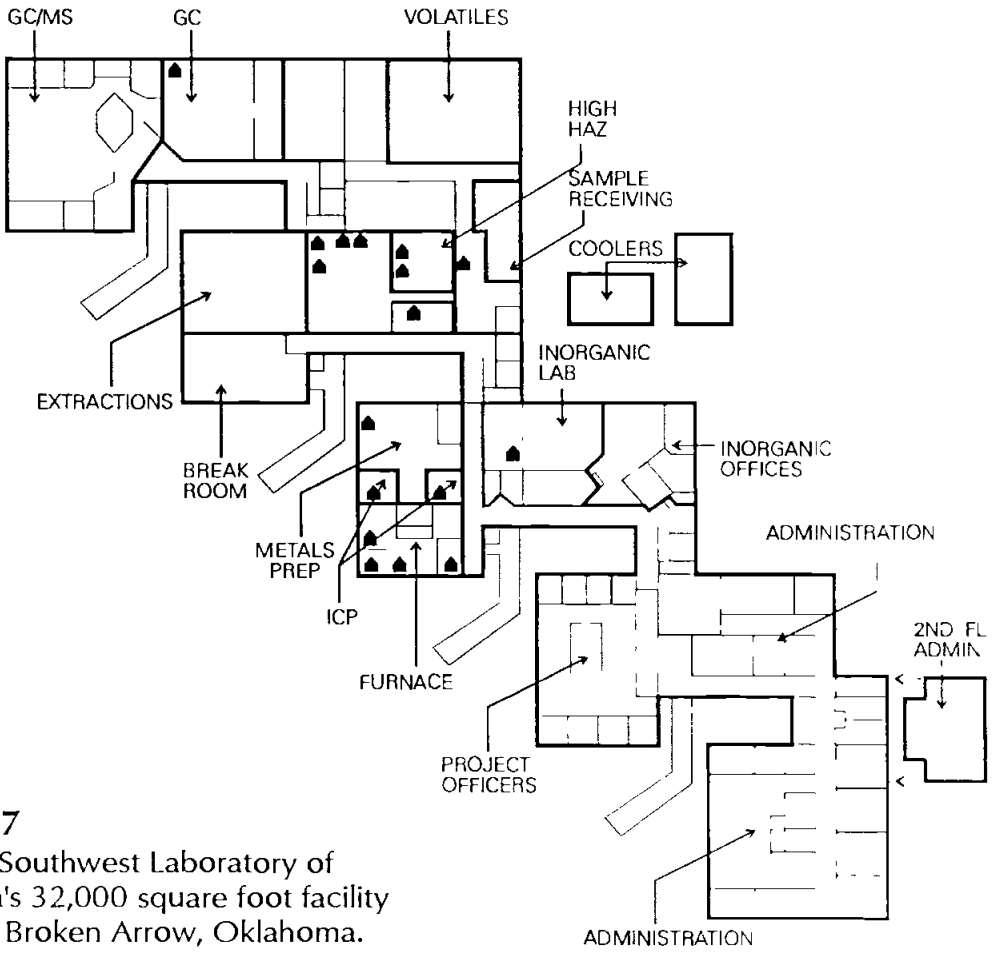


FIGURE 7
Layout of Southwest Laboratory of Oklahoma's 32,000 square foot facility located in Broken Arrow, Oklahoma.

PROJECT DESCRIPTION

This quality assurance project plan is designed to meet the QA/QC and analytical requirements of Burns & McDonnell and EPA. The project involves work on the USRGAFB.

The laboratory will perform analyses on aqueous, soil, sludge, oil, and other miscellaneous matrices collected by the client.

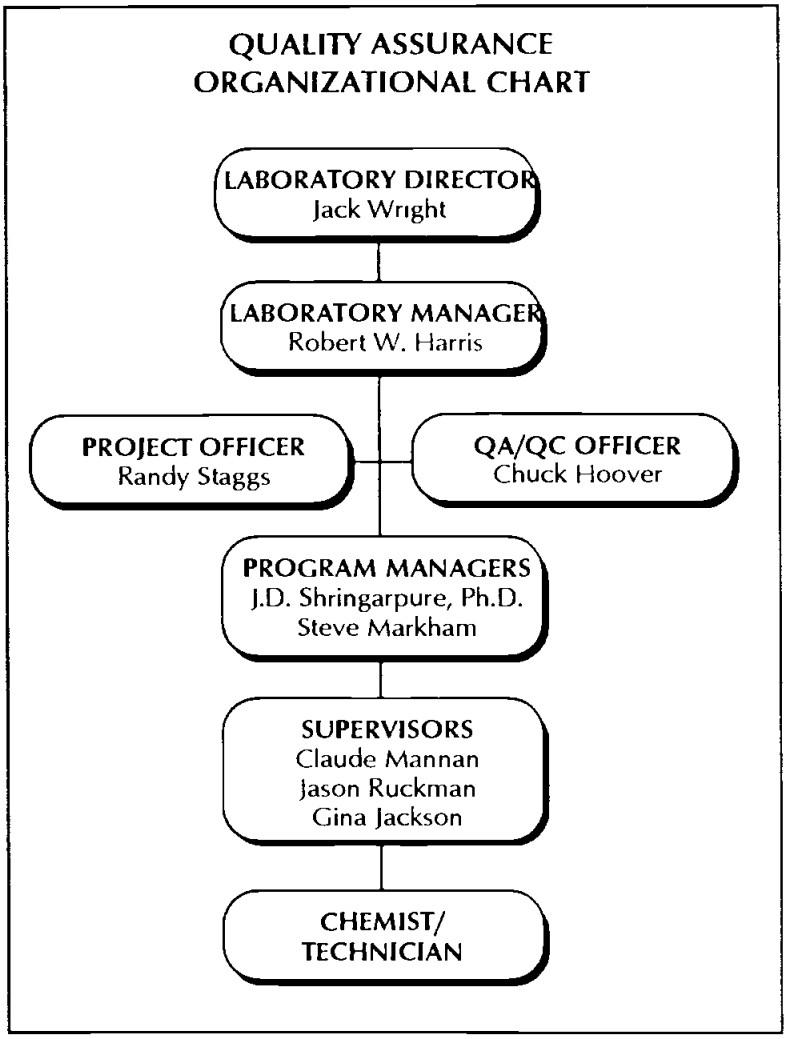
To minimize sample contamination, appropriate sample and shipping containers will be sent to the project site at the client's request. Client will supply Chain-of-Custody forms. Samples with completed labels and forms will be express shipped to the laboratory where the sample custodian will accept custody. The custodian will verify information on sample labels and Chain-of-Custody. Discrepancies or breakages will be noted on Chain-of-Custody and client will be notified. Samples will not be analyzed until the problem is resolved. Samples will be logged into the "Laboratory Information Management System" (LIMS) and Chain-of-Custody forms updated. Samples with control documents will be processed thru the analytical sequence.

Southwest Laboratory of Oklahoma's standard laboratory procedure is to retain samples for ninety days. Upon client's request, samples can be retained for up to 180 days. The laboratory will dispose of all residual sample material including samples containing environmentally hazardous constituents (or display hazardous characteristics) in an environmentally safe manner, mutually agreed upon by both parties.

Data reduction and validation will be performed in accordance with the referenced method or specific Standard Operating Procedures. Data reporting will be in tabular format to meet client requirements.

RESPONSIBILITIES AND AUTHORITIES

A Quality Assurance Program for an environmental laboratory requires the attention and commitment of both management and staff to be effective. The Quality Assurance effort at SWL is managed by the Quality Assurance Officer which reports directly to the Laboratory Manager. The Quality Assurance Officer operates independently from all areas generating analytical data to insure complete objectivity in the evaluation of laboratory operations.



The implementation of the Quality Assurance Program within each individual laboratory is the responsibility of the Program Manager. We believe that the success of SWL is dependent upon the continued commitment of all within the organization to a strong and viable Quality Assurance Program. The responsibilities and levels of authority within the organization are described below.

The Quality Assurance effort within SWL is directed by the Laboratory Manager who reports directly to the President of SWL. The Quality Assurance Officer also assists the Laboratory Manager in carrying out the responsibilities of the laboratory.

LABORATORY MANAGER

Responsibilities

The Laboratory Manager is responsible for:

- Developing and implementing a corporate Quality Assurance Program which assures that all data generated in SWL laboratories is scientifically

sound, legally defensible, and of known precision and accuracy

- Developing and implementing new Quality Assurance procedures within the corporation to improve data quality.
- Directing the Program Manager in the implementation of the SWL Quality Assurance Plan within individual sections.
- Interfacing on Quality Assurance issues for both clients and laboratory staff.
- Promoting sound Quality Assurance practices within the environmental regulatory and analytical communities.

Authority

The Laboratory Manager is the final authority on all issues dealing with data quality and has the authority to require that procedures be amended or discontinued, or analyses suspended or repeated. The authority of the Laboratory Manager comes directly from the President of SWL, to whom the Laboratory Manager reports.

PROGRAM MANAGERS

The supervisors and managers who direct the analytical work at each section are directly responsible for ensuring that all employees reporting to them are complying with the SWL Quality Assurance Plan.

Responsibilities

The Program Managers are responsible for:

- Actively supporting the implementation of the SWL Project Quality Assurance Plan within the laboratory.
- Maintaining accurate SOP's and enforcing their use in the laboratory.
- Maintaining a work environment which emphasizes the importance of data quality.
- Providing management support to the Quality Assurance Officer and the Laboratory Manager.
- Prescribing and monitoring Corrective Action.

Authority

The Program Managers of the laboratory have the authority to accept or reject data based on well-defined Quality Control criteria. In addition, Program Managers, with the approval of the Quality Assurance Officer, can accept data which falls outside the normal Quality Control limits if, in their judgment, there are technical reasons which warrant the acceptance of the data. These circumstances must be well documented and any need for Corrective Action identified by the incident must be defined and initiated.

CHEMIST/TECHNICIAN

All laboratory personnel involved in the generation and reporting of data have a responsibility to understand and follow the SWL Quality Assurance Plan.

The Chemists and Technicians are responsible for:

- Having a working knowledge of the SWL Quality Assurance Plan.
- Ensuring that all work is generated in compliance with the SWL Quality Assurance Plan.
- Performing all work according to written SOPs.
- Ensuring that all documentation related to their work is complete and accurate.
- Providing management with immediate notification of quality problems.

Authority

The chemists and technicians have the authority to accept or reject data based on compliance with well-defined Quality Control acceptance criteria. The acceptance of data which falls outside Quality Control criteria must be approved by laboratory management.

QUALITY ASSURANCE OFFICER

Responsibilities

The Quality Assurance officer is responsible for:

- Implementing SWL Quality Assurance policies.

- Monitoring the Quality Assurance Plan within the laboratory to ensure complete compliance with Quality Assurance objectives.
- Conducting in-house audits to identify potential problems and ensure compliance with written SOPs.
- Monitoring new personnel to verify quality of work.
- Performing statistical analyses of Quality Control data and establishing data bases which accurately reflect the performance of the laboratory.
- Monitoring corrective actions.
- Serving as in-house client representative on all project inquiries involving data quality issues.
- Monitoring the preparation and verification of analytical standards.
- Distributing current SOPs to the laboratory staff.
- Monitoring laboratory performance in the areas of holding times, turn-around times, and meeting contractual obligations.
- Preparing Quality Assurance project plans when needed.
- Assisting the Laboratory Manager in the writing of Quality Assurance manuals and procedures.

Key Personnel Experience

JACK WRIGHT, Laboratory Director
B.S., Chemical Engineering
GC/MS ..... 7 years
Purge and Trap ..... 6 years
Gas Chromatography ..... 12 years
Atomic Absorption ..... 12 years

ROBERT HARRIS, Laboratory Manager
B.S., Microbiology
GC/MS ..... 4 years
ICP ..... 1 year
Purge and Trap ..... 3 years
Gas Chromatography ..... 10 years
Atomic Absorption ..... 15 years
Sample Preparation ..... 15 years
Data Review ..... 15 years

CHUCK HOOVER, QA/QC Officer
B.A., Biology, (Chemistry Minor)
GC/MS ..... 2 years
Purge and Trap ..... 2 years
Gas Chromatography ..... 5 years
Atomic Absorption ..... 4 years
Sample Preparation ..... 8 years
Data Review ..... 6 years

STEVE MARKHAM, Inorganics Manager
B.S., Zoology
ICP ..... 7 years
Atomic Absorption ..... 11 years
Sample Preparation ..... 12 years
Data Review ..... 11 years
Inorganic Analysis ..... 13 years

J.D. SHRINGARPURE, Ph.D., Organics Program Mgr.
Ph.D., Organic Chemistry
GC/MS ..... 8 years
Purge and Trap ..... 8 years
Gas Chromatography ..... 13 years
Sample Preparation ..... 8 years
Data Review ..... 9 years

DARYL ALSTATT, Project Officer
B.A., Chemistry
Sample Preparation ..... 5 years
Data Review ..... 4 years
Inorganic Analysis ..... 5 years

RANDY STAGGS, Project Officer
B.S., Biology
Gas Chromatography ..... 3 years
Atomic Absorption ..... 5 years
Sample Preparation ..... 5 years
Data Review ..... 10 years
Inorganic Analysis ..... 15 years

MARK SMITH, Organics Data Manager
B.S., Chemistry
GC/MS ..... 5 years
Purge & Trap ..... 5 years
Sample Preparation ..... 1 year
Data Review ..... 3 years
Inorganic Analysis ..... 0.5 years

JASON RUCKMAN, AA Furnace Metals Supv.
B.S., Chemistry
ICP ..... 4 years
Atomic Absorption ..... 5 years
Sample Preparation ..... 4 years
Data Review ..... 4 years

GINA JACKSON, GC/MS Laboratory
B.S., Chemistry
GC/MS ..... 4 years
Purge & Trap ..... 1 year
Sample Preparation ..... 1 year

CLAUDE MANNAN, GC Laboratory Mgr.
M.S. , Chemistry
GC/MS ..... 12 years
Purge & Trap ..... 6 years
Gas Chromatography ..... 20 years
High Performance Liq. Chromatography ..... 20 years
Data Review ..... 7 years
Computer Programming ..... 20 years

LABORATORY CONTROL LIMITS

PARAMETER	METHOD	REFERENCE	ACCURACY%	PRECISION (MAX RPD) WATER/SOIL	COMPLETE-NESS
Volatile Organics	8010/8020	SW847			90%
• **Surrogate Spikes**					
cis-1,2-Dichloroethene			40-140		
p-chlorotoluene			40-140		
Bromofluorobenzene			60-130		
• **Matrix Spike/Duplicate (RPD)**				30	
Total Petroleum Hydrocarbons	418.1	EPA	75-125		90%
Anions (Cl^-,F^-,NO_3^-, etc.)	300.0	EPA	75-125	25	90%
Trace Metals (ICP)*	200.7/6010	EPA/SW846			90%
Antimony			75-125	20	
Barium			75-125	20	
Beryllium/Cadmium			75-125	20	
Calcium			75-125	20	
Chromium			75-125	20	
Cobalt			75-125	20	
Copper			75-125	20	
Iron			75-125	20	
Magnesium			75-125	20	
Manganese			75-125	20	
Nickel			75-125	20	
Potassium			75-125	20	
Silver			75-125	20	
Sodium			75-125	20	
Tin			75-125	20	
Vanadium			75-125	20	
Zinc			75-125	20	
Trace Metals (Furance AA)*	7000 Series	SW-846			90%
Arsenic			75-125	20	
Lead			75-125	20	
Selenium			75-125	20	
Thallium			75-125	20	
Mercury (CV)	7470	SW-846	75-125	20	90%

SAMPLING PROCEDURES

For this project SWL's sampling procedures are not applicable

CHAIN-OF-CUSTODY & RECORDS

Field Sampling Operations will not be managed by Southwest Laboratory for this project.

Keeping in mind that all information received is important, SWLO's staff uses an extensive record system. It begins with a sample custodian's numbering of each sample before it leaves the receiving area. The sample custodian is responsible for the sample while it is in the receiving area. The identification number is entered in a computerized Laboratory Information Management System, (LIMS), with other pertinent information such as client name and number, source of sample, sampling date, and date and time the sample is received. All associated paperwork is retained in a case file (chain-of-custody sheets, extraction logs, traffic reports, instrument logs, etc.). See Figures 6.1 through 6.13.

An analysis sheet, which functions as an instruction sheet and report form, is prepared at the same time and contains, in addition to the above information, lab identification number, parameters to be determined, methods to be used, and special instructions. Special instructions are highlighted at the bottom of the sheet (i.e. holding time less than five days, RUSH sample, etc.). One analysis sheet accompanies each sample.

Internal Chain-of-Custody sheets are also produced by the LIMS System at the time of sample log in. These consist of two forms, a sample tracking sheet and an extract tracking sheet (Figures 6.2 & 6.3).

Sample tracking sheets follow the sample from storage check out through extractions and into archive. Extract tracking sheets follow the extracts from the sample preparation lab through the Instrumentation Laboratories and back to archive.

Both internal Chain of Custodies are placed in the case file along with other pertinent paper work.

The identification number is transcribed permanently on the label of the bottle or bottles which comprise one sample. Each bottle of a set constituting one sample is also permanently labeled with the type of preservative it contains.

Samples are separated by type, i.e., VOA, BNA, Pesticides, etc., then placed on carts in sequential order received, and taken to the laboratory by the laboratory supervisor. This ensures efficiency and keeps the supervisor apprised of samples.

QA/QC samples are split out at this time; One sample in every twenty is analyzed (in addition to regular analysis) as a Matrix Spike & Matrix Spike Duplicate.

The manager or supervisor assigns the analysis of perishable samples immediately. Other samples, at the direction of the lab supervisor, either remain on carts or are stored under refrigeration. The analysts are responsible for the orderly movement of samples through the laboratory so that all determinations are completed in minimum time.

The manager receives the analysis sheet and from its assigns in writing the daily schedule for the analyst. Analysis sheets are then placed in numeric order in the area where work is to be performed.

An extraction logbook, permanently bound and numbered is used during sample preparation (figure 6.4). It contains notes, descriptions, and test methods used. A typical entry in the notebook includes the date of analysis, permanent identification number, parameter being determined, the method used, aliquots taken, Surrogate/Matrix Spiking Solutions used, solvent amounts, cleanup used, and final process volume.

The supervisor is responsible for checking entries in the extraction log. The supervisor signs and dates the extraction notebook page and sends it along with the extracts to the Instrumentation Laboratory.

All pertinent information, identification number, client name and number, date received, date completed, and parameter results are transferred to a report. A copy of the computer generated report is sent to the client. Both the original sheet and original typed report are filed according to client name or report number; the date reported is recorded in the logbook.

Retrieval of data is possible by either client name and final report number, date received, or permanent identification number. Through this system of record keeping, the original data can be easily located.

All records, including analysts' retired extraction log books, are kept for three years.

When the requested parameters have been determined and results reviewed by the supervisor and laboratory manager, the manager directs that the samples be stored under appropriate conditions. Unless otherwise requested by the client, sample and bottles are returned to the client four weeks after the analysis report is issued.

SWOK/AATS

Date: 04/10/91

1700 ALBANY SUITE C
[illegible] OK 74012

Client:

SID #	DATE	DESCRIPTION	MA	MC	TEST	PRI	DUE	DESCRIPTION	RESULTS	ANALYSTS	DATE/TIME
9580.01	04/05/91	CEB-51 (CASE#16181)	W	3	MS310	3	05/04/91	VOA - CLP		BLC	04/08/91
9580.02	04/05/91	CEB-52 (CASE#16181)	S	7	MS310	3	05/04/91	VOA - CLP			
					MS510	3	05/04/91	SEMIVOL CLP			
								EXTRACTION			
9580.03	04/05/91	CEB-53 (CASE#16181)	S	3	MS310	3	05/04/91	VOA - CLP			
					MS510	3	05/04/91	SEMIVOL CLP			
								EXTRACTION			
9580.04	04/05/91	CEB-55 (CASE#16181)	W	4	GC810	3	05/04/91	PEST/PCB CLP			
								EXTRACTION		TR	05-APR-91
					MS310	3	05/04/91	VOA - CLP		BLC	04/08/91
					MS510	3	05/04/91	SEMIVOL CLP			
								EXTRACTION		TR	05-APR-91
9580.05	04/05/91	CEB-56MS/MSD	S	6	GC810	3	05/04/91	PEST/PCB CLP			
								EXTRACTION			
					MS310	3	05/04/91	VOA - CLP			
					MS510	3	05/04/91	SEMIVOL CLP			
								EXTRACTION			
9580.06	04/05/91	CEB-57 (CASE#16181)	S	3	GC810	3	05/04/91	PEST/PCB CLP			
								EXTRACTION			
					MS310	3	05/04/91	VOA - CLP			
					MS510	3	05/04/91	SEMIVOL CLP			
								EXTRACTION			
9580.07	04/05/91	CEB-58 (CASE#16181)	S	3	GC810	3	05/04/91	PEST/PCB CLP			
								EXTRACTION			
					MS310	3	05/04/91	VOA - CLP			
					MS510	3	05/04/91	SEMIVOL CLP			
								EXTRACTION			

FIGURE 6.1
Sample Work Sheet

INTERNAL CHAIN OF CUSTODY
SAMPLE TRACKING SHEET
SDG NUMBER :
FRACTION : VOA
MATRIX : Soil

SWOK/AATS

DATE LOGGED-IN/ ANALYST	SAMPLE #	CASE/SAMPLE ID	NC	DATE LOGGED-OUT FOR ANALYSIS/ ANALYST	ARCHIVE DATE/ ANALYST	DATE DISCARDED/ ANALYST
04/05/91	5580.02	CEB-52 (CASE816181)	3			
04/05/91	5580.03	CEB-53 (CASE816181)	3			
04/05/91	5580.05	CEB-56MS/MSD	6			
04/05/91	5580.06	CEB-57 (CASE816181)	3			
04/05/91	5580.07	CEB-58 (CASE816181)	3			
04/05/91	5580.08	CEB-59 (CASE816181)	3			
04/05/91	5580.09	CEB-60 (CASE816181)	3			

INTERNAL CHAIN OF CUSTODY
EXTRACT TRACKING SHEET
SDG NUMBER :
FRACTION : PEST
MATRIX : Soil

SWOK/AATS

REFRIG. #2 DATE LOGGED-IN/ ANALYST	SAMPLE #	CASE/SAMPLE ID	EXT. VOL.	DATE TO ANALYSIS/ ANALYST	ARCHIVE DATE/ ANALYST	DATE DISCARDED/ ANALYST
	5580.05	CEB-56MS/MSD				
	5580.06	CEB-57 (CASE816181)				
	5580.07	CEB-58 (CASE816181)				
	5580.08	CEB-59 (CASE816181)				

ABOVE:
FIGURE 6.2
Internal Sample Chain-of-Custody, Volatiles

BELOW: FIGURE 6.3
Internal Extract Chain-of-Custody, Pesticides

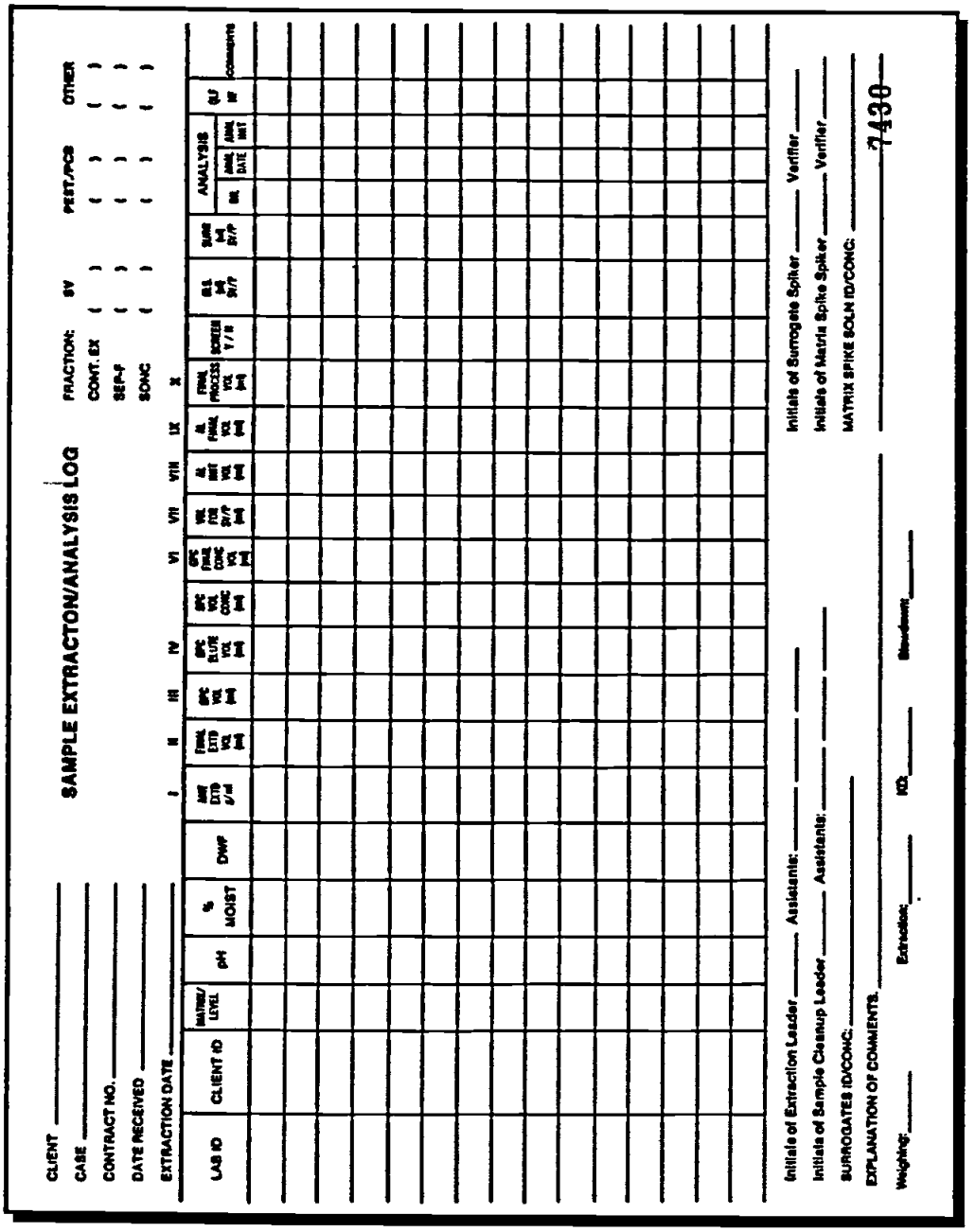
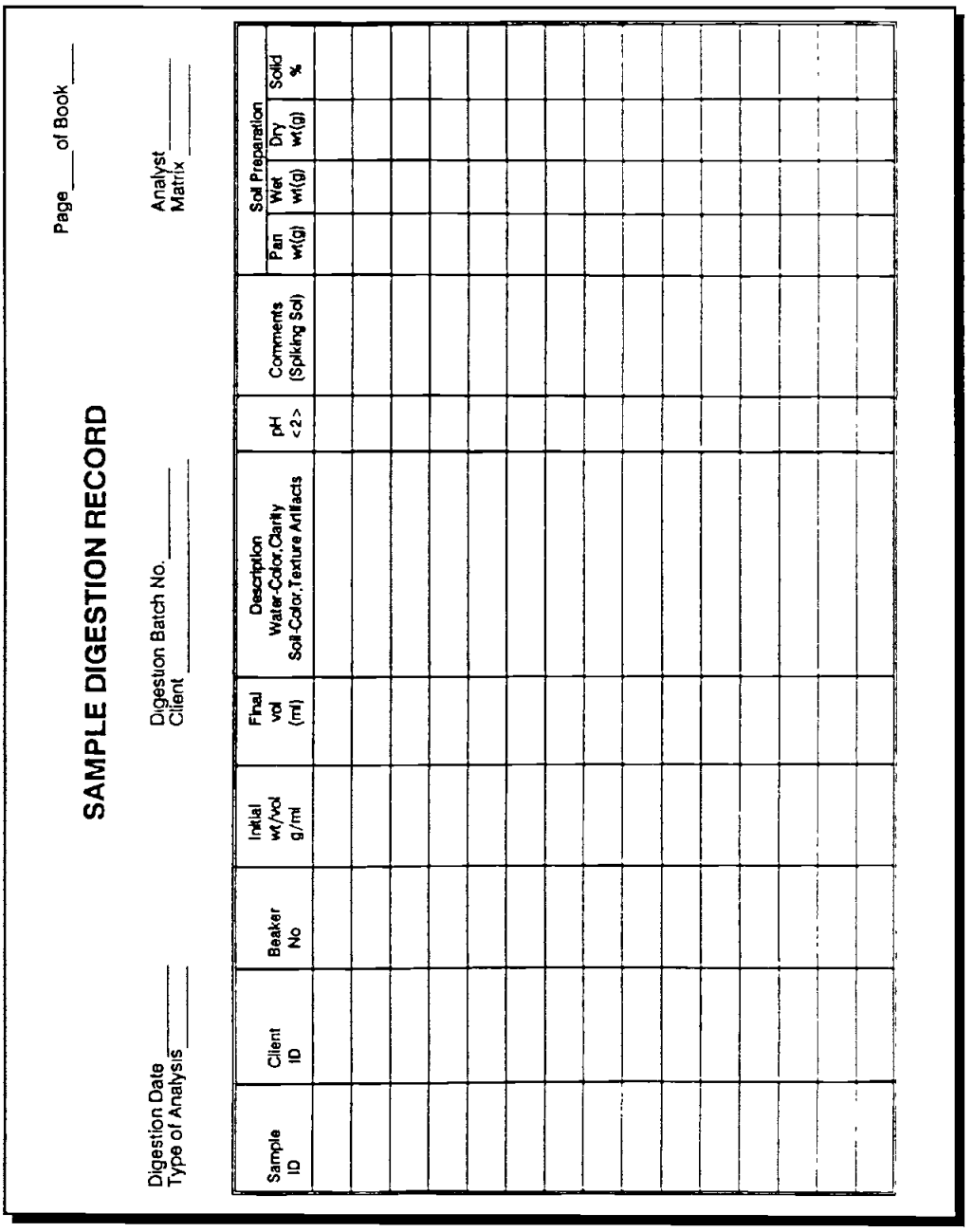


FIGURE 6.4
Sample Extraction/Analysis Log



SAMPLE DIGESTION RECORD

Page___ of Book___

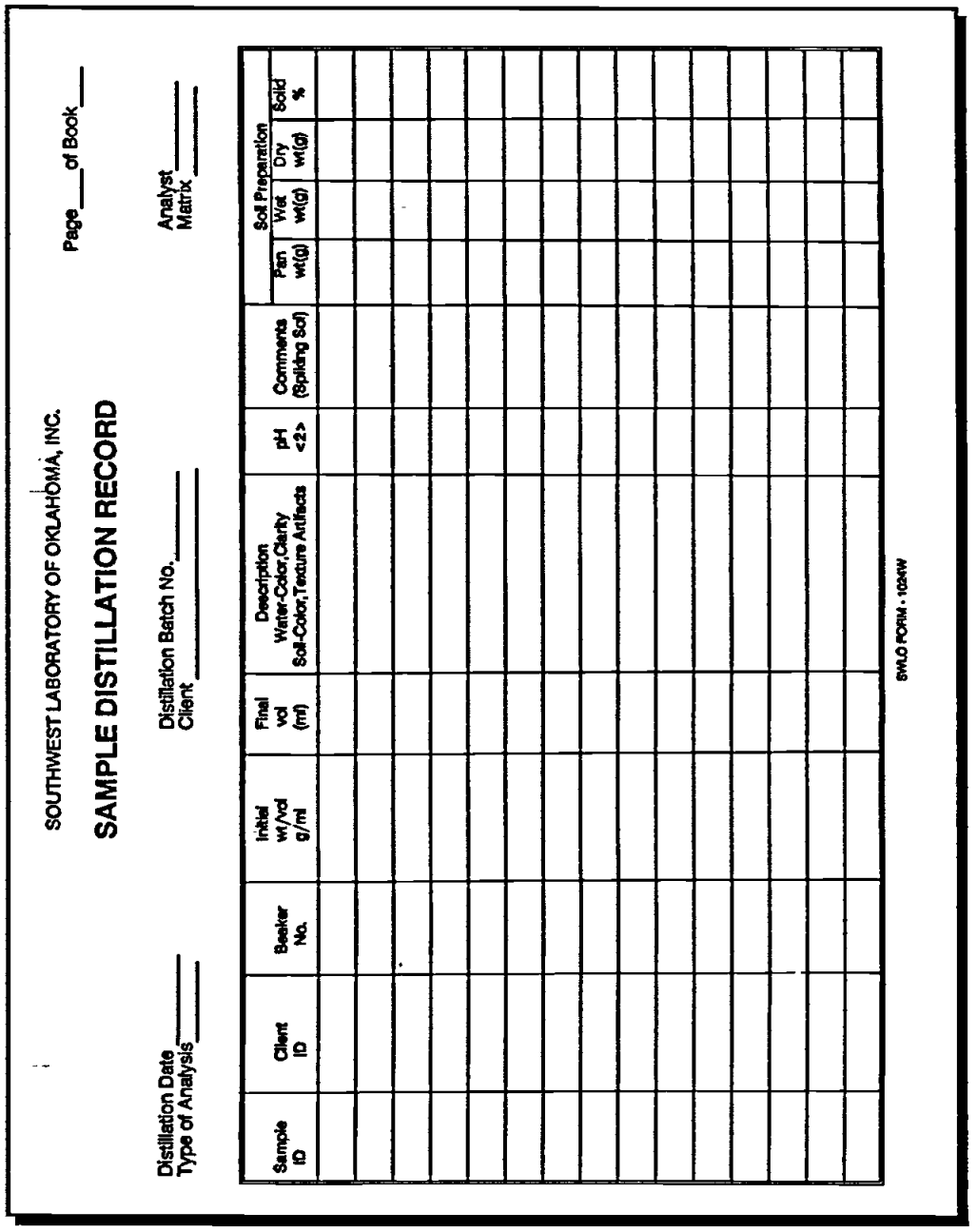
Digestion Batch No._____
Client _____

Analyst _____
Matrix _____

Digestion Date _____
Type of Analysis_____

Sample ID	Client ID	Beaker No	Initial wt/vol g/ml	Final vol (ml)	Description Water-Color,Clarity Soil-Color,Texture Artifacts	pH <2>	Comments (Spiking Sol)	Soil Preparation			
								Pan wt(g)	Wet wt(g)	Dry wt(g)	Solid %

FIGURE 6.5
Sample Digestion Record



SOUTHWEST LABORATORY OF OKLAHOMA, INC.

Page ___ of Book ___

SAMPLE DISTILLATION RECORD

Distillation Batch No. _____
Client _____

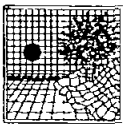
Analyst _____
Matrix _____

Distillation Date _____
Type of Analysis _____

Sample ID	Client ID	Beaker No.	Initial wt/vol g/ml	Final vol (ml)	Description Water-Color,Clarity Soil-Color,Texture Artifacts	pH <2>	Comments (Spiking Sol)	Soil Preparation Pan wt(g)	Wet wt(g)	Dry wt(g)	Solid %

SW.O FORM - 1024W

FIGURE 6.6
Sample Distillation Record



VOLATILES GC/MS RUN LOG

SWOK/AATS

TUNE FILE ID _____ METHOD FILES _____

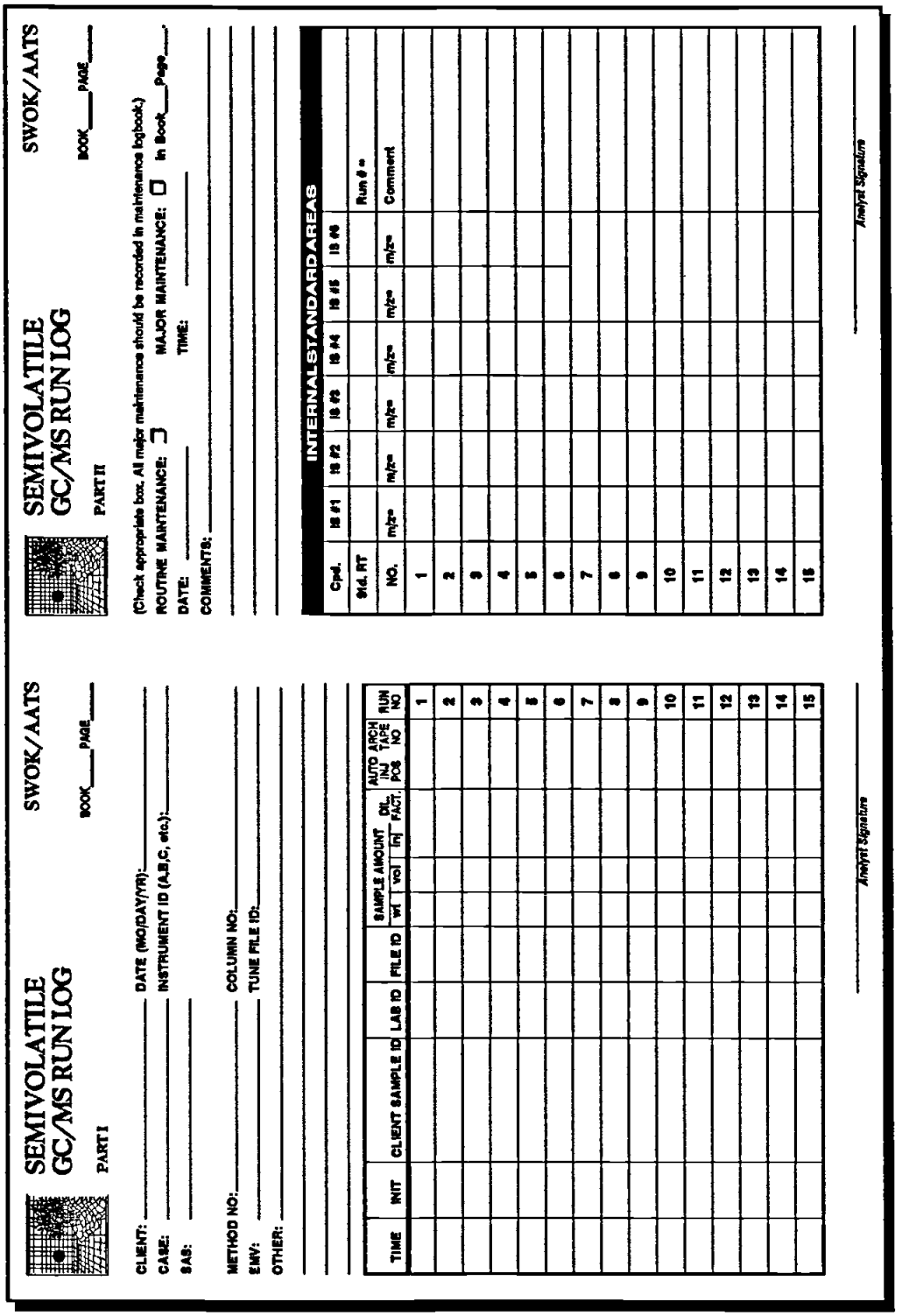
INSTRUMENT: _____ DATE _____

CB/D FILE ID _____ ARCHIVE TAPE #: _____

Time	File	SWOK/AATS Sample ID	Client ID	Vol.	IS Area	Surr Rec.	Lin. Chk	Comments	OK/ RR

Analyst Signature

FIGURE 6.7
Volatiles GC/MS Run Log



SEMIVOLATILE GC/MS RUN LOG — PART I — SWOK/AATS

BOOK___ PAGE___

CLIENT: _____ DATE (MO/DAY/YR):_____

CASE: _____ INSTRUMENT ID (A,B,C, etc.):_____

SAS: _____

METHOD NO:_____ COLUMN NO:_____

ENV: _____ TUNE FILE ID:_____

OTHER: _____

TIME	INIT	CLIENT SAMPLE ID	LAB ID	FILE ID	SAMPLE AMOUNT wt	SAMPLE AMOUNT vol	SAMPLE AMOUNT inj	DIL. FACT.	AUTO INJ POS	ARCH TAPE NO	RUN NO
											1
											2
											3
											4
											5
											6
											7
											8
											9
											10
											11
											12
											13
											14
											15

Analyst Signature _____

SEMIVOLATILE GC/MS RUN LOG — PART II — SWOK/AATS

BOOK___ PAGE___

(Check appropriate box. All major maintenance should be recorded in maintenance logbook.)

ROUTINE MAINTENANCE: ☐ MAJOR MAINTENANCE: ☐ In Book___ Page___

DATE: _____ TIME: _____

COMMENTS: _____

INTERNAL STANDARD AREAS

Cpd.	IS #1	IS #2	IS #3	IS #4	IS #5	IS #6		
Std. RT								
NO.	m/z=	m/z=	m/z=	m/z=	m/z=	m/z=	Run # =	Comment
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								
11								
12								
13								
14								
15								

Analyst Signature _____

FIGURE 6.8
Semivolatiles GC/MS Run Log

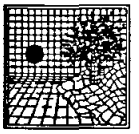
SWOK/AATS PESTICIDE - PCB INSTRUMENT_____
CASE NO._____ INJ.VOL._____
SDG NO._____ GC RUN LOG METHOD FILENAME_____

DATE	TIME	CLIENT I.D.	SWOK/AATS SAMPLE I.D.	FILE NAME	COMMENTS	INT.

Verification Signature Analyst Signature

Date Date

FIGURE 6.9
Pesticides-PCBs GC Run Log



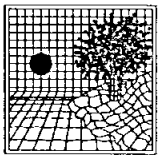
ICP RUN LOG

ANALYST: _____ DATE _____

INSTRUMENT: _____ CALIBRATION STD: SOURCE _____

Client ID	SWLO ID	Time	Percent Recovery	Dilution Factor	Analytes (circle)
					Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, V, Zn
					Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, V, Zn
					Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, V, Zn
					Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, V, Zn
					Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, V, Zn
					Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, V, Zn
					Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, V, Zn
					Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, V, Zn
					Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, V, Zn
					Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, V, Zn
					Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, V, Zn
					Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, V, Zn
					Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, V, Zn
					Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, V, Zn
					Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, V, Zn
					Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, V, Zn
					Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, V, Zn
					Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, V, Zn
					Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, V, Zn
					Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, V, Zn
					Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, V, Zn
					Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, V, Zn
					Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, V, Zn
					Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, V, Zn

FIGURE 6.10
ICP Run Log



FURNACE RUNLOG

AATS / SWOK

WAVELENGTH: **INTEGRATION TIME:** **INSTRUMENT:** **ANALYST/DATE.**

Arsenic = 193 70
Lead = 283 30
Selenium = 196 00
Thallium = 276 00

5.0 Sec.

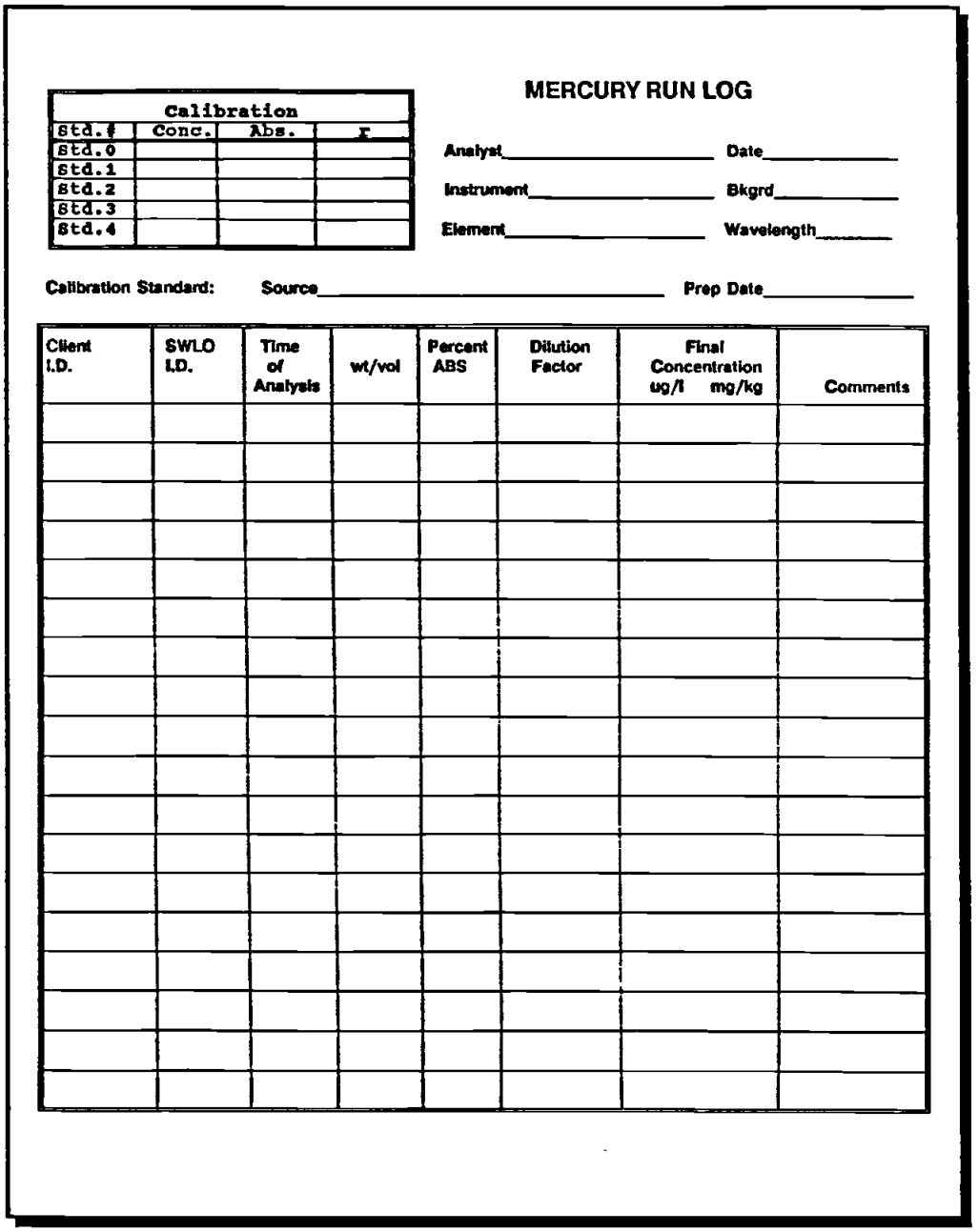
BKGRND CORRECTION: **DATA FILE:** **ELEMENT RAN:**

AS PB SE TL

#	Lab ID #	Client ID #	Used	Used
1.				
2.				
3.				
4.				
5.				
6.				
7.				
8.				
9.				
10.				
11.				
12.				
13.				
14.				
15.				
16.				
17.				
18.				
19.				
20.				

#	Lab ID #	Client ID #	Used	Used
21.				
22.				
23.				
24.				
25.				
26.				
27.				
28.				
29.				
30.				
31.				
32.				
33.				
34.				
35.				
36.				
37.				
38.				
39.				
40.				

FIGURE 6.11
Furnace Run Log



MERCURY RUN LOG

Calibration			
Std.#	Conc.	Abs.	r
Std.0			
Std.1			
Std.2			
Std.3			
Std.4			

Analyst_____ Date_____

Instrument_____ Bkgrd_____

Element_____ Wavelength_____

Calibration Standard: Source_____ Prep Date_____

Client I.D.	SWLO I.D.	Time of Analysis	wt/vol	Percent ABS	Dilution Factor	Final Concentration ug/l mg/kg	Comments

FIGURE 6.12
Mercury Run Log

SOUTHWEST LABORATORY OF OKLAHOMA, INC.

CN^- ANALYSIS LOGSHEET

Analyst: _____ EPA Case No.: _____

Distillation Date _____ SDG No.: _____

Lab Batch No.: _____

EPA Sample #	Lab Sample #	Absorbance	(mg)	Dilution Factor	Initial Weight (g) Volume (ml)	Final Concentration ug/l mg/kg

Distillate Transfer

Removed from Storage	Returned to Storage
Area #. _____	Area #. _____
By: _____	By: _____
On: _____	On _____

Received by_____Date_____ Logbook No : _____

Page No.: _____

FIGURE 6.13
CN^- Analysis Log Sheet

CALIBRATION/STANDARDIZATION

CALIBRATION PROCEDURES AND FREQUENCIES

Calibration is the process for determining the correctness relative to physical or chemical standards used or assigned values in scales of measuring instruments. It establishes a reproducible reference point to which all sample measurements can be correlated.

LABORATORY INSTRUMENTATION

Inorganic Chemistry Section

Atomic Absorption Spectrophotometer Systems (AAS)/Inductively Coupled Argon-Plasma Emission Spectrophotometer (ICAP)

For AAS systems (i.e., flame AA, graphite furnace AA and ICP), the instruments are calibrated daily, and each time the instrument is set-up. Appendix III lists the instrument operating parameters employed in the analysis.

Calibration standards are prepared fresh before each analysis and are discarded after use. These calibration standards are checked for traceability using NBS reference standards or EPA QC solutions. Appendix IV contains a list of NBS and EPA reference standards available for laboratory use.

The following QA/QC requirements are employed for the AA calibration:

a. Initial Calibration Verification (ICV)

- The accuracy of the initial instrument calibration must be verified and documented for every analyte by the analysis of an Initial Calibration Verification solution (ICVS - i.e., a NBS, SRM or EPA QC solution). When measurements exceed the control limits of the ICVS or within the target range values supplied by the QC solution, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration reverified.

- If the ICVS is not available, or if a certified solution of an analyte is not available from any source, analyses shall be conducted on an independent

standard at a concentration other than that used for calibration, but within the calibration range. (See Appendix IV for Criteria and Guidelines for Standard Traceability)

- The ICVS must be run at each wavelength used for analysis.

b. Calibration Blank

- Must be analyzed each time the instrument is calibrated.
- Must be analyzed at the beginning and the end of the run, and at a frequency of 10% during the run. It is also analyzed after a standard run or after a contaminated sample run to check for carry-over contamination.
- Blank results are to be reported down to the instrument detection limits (IDL).
- If the result is greater than method detection limit (MDL), the analysis is terminated, problem corrected, the instrument recalibrated and calibration reverified.

c. Continuing Calibration Verification (CCV)

- CCV must be performed for each analyte at a frequency of 10% or every two hours during analysis, whichever is more frequent.
- CCV must be also analyzed for each analyte at the beginning and at the end of the analysis.
- The analyte concentration in the CCV must be near the mid-range of the calibration curve.
- The same calibration standard must be used throughout the analysis for a particular case.
- One of the following standards can be used for continuing calibration verification:

    1. EPA Solution
    2. NBS SRM
    3. In-house prepared solution from an independent standard

- If the CCV results exceed the specified control limits (i.e., 95% confidence limits of the true values or the given target range values), the instrument must be recalibrated and the preceding 10 samples or less reanalyzed for the analytes affected.

d. ICP Interference Check Sample Analysis (ICS)

- To determine if interelement and background correction is required, ICS is analyzed at the beginning and end of each sample analysis run (minimum of 2 times for every 8 hours).

- Results must fall within the control limits of ± 20% of the EPA supplied true values for the analytes included in the ICS. (See Table III-5.1 for list of analytes and interferants in the ICP check sample). If not within the control limits, the analysis is terminated, the problem is corrected, the instrument is recalibrated, and the samples are reanalyzed.

- If an EPA ICP check sample is not available, an independent ICP check sample is prepared with the interferant and analyte concentration at the levels specified in Table III-5.1. The mean value and the standard deviation is established by initially analyzing the prepared check sample at least 5 times for each parameter. Control limits are then established for the in-house prepared solution. It must fall within ± 20% of the mean value.

If an interference cannot be resolved successfully, a standard addition technique will be used for both AA and ICAP. Standards of the analytes will be added to the duplicate sample and the concentration of the analyte(s) can be determined by difference.

The following requirements are employed when using a method of standard addition for graphite furnace analysis:

- Data must be within the linear range determined by the calibration curve.

- The sample and the three spikes must be analyzed consecutively.

- Only single injections are required.

- Spikes should be prepared such that:

  1. Spike 1 is approximately 50% of the sample absorbance.

2. Spike 2 is approximately 100% of the sample absorbance.

3. Spike 3 is approximately 150% of the sample absorbance.

Organic Processing Section

Gel Permeation Chromatograph (GPC)

The following procedure is employed for the calibration of the GPC system:

- Packing the column - Place 70 grams (g) of Bio Beads SX-3 in a 400 milliliter (mL) beaker. Cover the beads with methylene chloride and allow the beads to swell overnight before packing the column. Transfer the swelled beads to the column and start pumping solvent through the column, from bottom to top, at 5.0 mL/minute. After approximately 1 hour, adjust the pressure on the column to 7-10 psi and pump an additional 4 hours to remove air from the column. Adjust the column pressure periodically as required to maintain 7-10 psi.

- Prepare the GPC calibration solutions as follows: (1) Corn oil - Add 200 mg corn oil to sufficient amount of methylene chloride to attain a final volume of 1 mL; (2) phthalate-phenol - Add 4.0 mg each of Bis(2-ethylhexylphthalate) and pentachlorophenol to sufficient amount of methylene chloride to attain a final volume of 1 mL.

- Calibration of the column - Load 5 mL of the corn oil solution into sample loop No. 1 and 5 mL of the phthalate-phenol solution into loop No. 2. Inject the corn oil and collect 10 mL fraction (i.e., change fraction at 2-minute intervals) for 36 minutes. Inject the phthalate-phenol solution and collect 15 mL fraction for 60 minutes. Determine the corn oil elution pattern by evaporation of each fraction to dryness followed by a gravimetric determination of the residue.

- Analyze the phthalate-phenol fractions by GC/FID on the DB-5 capillary column.

- Plot the concentration of each component in each fraction versus total eluent volume (or time) from the injection points. Choose a "dump time" which allows $\geq$85% removal of the corn oil and $\geq$ 85% recovery of the

bis(2-ethylhexyl)-phthalate.

- Choose the "collect time" to extend at least 10 minutes after the elution of pentachlorophenol. Wash the column at least 15 minutes between samples.

- Typical parameters selected are: Dump time = 30 minutes (150 mL); collect time = 36 minutes (180 mL); and wash time = 15 minutes (75 mL).

General Laboratory Equipment

Balances are calibrated before every use with standard Class-S calibration weights and are calibrated annually by a licensed specialist. The pH/specific-ion meters are calibrated before each use with a minimum of three standard solutions. (See Appendix IV, Standard Operating Procedures for pH meter, balances, and conductivity meter for discussion)

Chromatography Section

Gas Chromatographs (GC)

Injection of secondary standards, validated by the use of EPA or NBS reference standards, are used to adjust the sensitivity and selectivity of the analytical system for each compound being analyzed. The system is calibrated by preparing standards at a minimum of three concentration levels for each analyte. The low-level standard is at or near the established detection limit. The medium- and high-level standards are at concentrations that correspond to the expected range of concentrations found in the samples. These standards will define the working range of the GC detector. (See Appendix IV, Traceability and List of EPA and NBS reference standards available)

The results of standard calibrations (low, medium, and high ranges) for each analyte are tabulated with respect to response versus concentration. The ratio between response and concentration, known as the response factor (RF), can be used to prepare a calibration curve for each compound. It is expressed in an equation as:

$$RF = \frac{\text{Area response of analyte}}{\text{Concentration of analyte in the standard}}$$

The following criteria are employed for the GC linearity calibration:

a. Initial Calibration Verification

- % Relative Standard Deviation (RSD) cut-off for RF = ± 35% for all analytes except for problematic (i.e., poor responder) analytes (e.g., gases, endrin, methoxychlor, endrin aldehyde). RSD is calculated as:

$$\% \text{ RSD} = \frac{s \times 100}{\text{average RF of the individual analytes in the standard solution}}$$

where s = standard deviation of the RF of each analyte
and where Average RF = mean of the RF of the analyte

- % RSD cut-off for problematic compounds = ± 50%

- If 20% of the analytes in the % RSD determination of the linearity standard check is greater than 35%, then linear regression or a straight-line curve is used to determine linearity. (Alternatively, there is an option to use the quadratic equation or a point-to-point curve if the correlation coefficient for the linear regression is less than 0.995.)

- If the compounds are still out of criteria after using linear regression, or the quadratic equation, or a point-to-point curve, reanalyze the standard concentration that is of suspect and then recalculate % RSD.

- If the compounds are still out of criteria after such reanalysis of the suspected standard, corrective and/or preventive maintenance should be performed to check the system. A new set of initial calibration curves is to be analyzed.

b. Continuing Calibration Verification

- Percent Difference (% D) cut-off for RF = ± 35% for all analytes except for problematic (i.e., poor responder) analytes (e.g., gases, endrin, methoxychlor, endrin aldehyde). % D is calculated as:

$$\% \text{ D} = \frac{(\text{average RF} - \text{RF (continuing calibration standard)}) \times 100}{\text{mean of average RF and RF (continuing calibration standard)}}$$

- % D cut-off problematic compounds = ± 50%

- If 20% of the analytes in the % D determination of the standards for linearity check have values greater than 35%, then linear regression or a straight-line curve is used to determine linearity. (Alternatively, there is an option to use the quadratic equation or a point-to-point curve if the correlation coefficient for the linear regression is less than 0.995.)

- If the compounds are still out of criteria after using linear regression, the quadratic equation, or a point-to-point curve, reanalyze the continuing calibration standard concentration and recalculate % D.

- If the compounds are still out of criteria after such reanalysis, corrective and/or preventive maintenance should be performed to check the system.

- A new set of initial calibration curves is to be analyzed.

c. The continuing calibration standard is analyzed every 12 hours during sample analysis.

d. A solvent blank using solvent (Pesticide grade) suitable for the detector is used to check system contamination and is also analyzed after a standard run or after a contaminated sample has been analyzed to check for carry-over contamination.

NOTE: Cut-off criteria for initial calibration and continuing calibration are interim guidelines. The GC laboratory will set new criteria based on historical data obtained from previous calibration standards used for the various analytical methods.

High Performance Liquid Chromatographs (HPLC)

The system is calibrated by preparing standards at a minimum of three concentration levels for each analyte. The low-level standard is at or near the established detection limit. The medium- and high-level standards are at concentrations that correspond to the expected range of concentrations found in the samples. These standards will define the working range of the HPLC. Continuing calibration is analyzed after every 10 samples and at the end of run. Criteria for % RSD for the initial calibration is within 10% or a linear regression or a straight-line curve is used to determine linearity. If the correlation coefficient is less than 0.995, corrective and/or preventive maintenance is done, and a new set of calibration curves is analyzed. Criteria for % D for continuing calibration is within 5% or a new standard is analyzed and % D recalculated.

Ion Chromatographs (IC)

The calibration procedure for the system is the same as the HPLC.

Mass Spectrometry Section

Procedures for calibration and instrument tuning for sensitivity and selectivity are somewhat similar to those for gas chromatography method. The primary difference between GC and GC/MS methods is concerned with the validation of the mass spectrometer as the detector. GC detectors generally operate by sensing a change in an electrical field (e.g., GC-EC, GC-FID, Hall); whereas, mass spectrometers sense a change in charge with reference to the mass of the compound. Further, the charge molecule ion will fragment reproducibly into an array of ions. The result is a characteristic mass spectrum of the compound. The first step in the calibration of the GC/MS system is to demonstrate the ionization and fragmentation of standard mass spectral tuning compounds. This is accomplished, as well as a sensitivity check, with the use of two EPA-specified compounds injected at concentration near the instrument detection limit. Those compounds are: 4-Bromofluorobenzene (BFB) for volatiles and Decafluorotriphenylphosphine (DFTPP) for semivolatiles. These standards are run daily to validate the GC/MS system tune. (See Tables III-5.2 & III-5.3, for Tune Criteria)

Calibration of the GC/MS, like that of GC calibration, is established and validated by the injection of EPA traceable standards at a minimum of three concentration levels over the range of likely sample concentrations. An internal calibration procedure is used: in addition to surrogate recovery compounds, sample extracts are spiked with internal calibration standards that span the retention time range of the analytes of interest. The concentration of the analytes is calculated with reference to the RF of the internal standards for each sample. RF is defined as:

$$RF = \frac{(A_x)(C_{is})}{(A_{is})(C_x)}$$

where A_x = area of the characteristic ion for the measured compound
A_{is} = area of the characteristic ion for the specific internal standard
C_{is} = concentration of the internal standard (ng/ul)
C_x = concentration of the measured compound (ng/ul)

Further precision, accuracy, and continuing calibration are demonstrated with the use of repeated analyses of spiked and duplicate spiked samples and EPA check samples. Reagent blanks are analyzed in each batch of semivolatile analyses and are analyzed daily for volatile organic analyses.

The QA/QC requirements employed for the GC/MS calibration are discussed in the following subsections.

Instrument Tuning

- Each EPA-CLP tune criteria for BFB and DFTPP are used before any sample analysis for VOA and BNA. (See Tables 4.2 and 4.3, BFB and DFTPP Tune Criteria.)

- A tune criteria is required every 12 hours during sample analysis.

Initial Instrument Calibration

- All compounds of interest are analyzed using a 5 pt. initial calibration. The validity of the calibration is based on specific criteria for certain compounds. These compounds are calibration check compounds (CCC) and system performance check compounds (SPCC).

- BNA and VOA CCC are:

BNA CCC	VOA CCC
Phenol	Vinyl Chloride
1,4-Dichlorobenzene	Chloroform
2-Nitrophenol	1,2-Dichloropropane
Hexachlorobutadiene	Toluene
4-Chloro-3-Methylphenol	Ethylbenzene
2,4,6-Trichlorophenol	
Acenaphthene	
N-Nitrosodiphenylamine	
Pentachlorophenol	
Fluoranthene	
Di-n-octyl phthalate	
Benzo(a)pyrene	

% RSD is calculated for all compounds. It should be within $\pm$ 30% for all CCC compounds and should be within $\pm$ 30% for all other compounds.

- BNA and VOA SPCC are:

BNA SPCC	VOA SPCC
N-Nitroso-di-n-Propylamine	Chloromethane
Hexachlorocyclopentadiene	1,1-Dichloroethane
2,4-Dinitrophenol	Bromoform
4-Nitrophenol	1,1,2,2-Tetrachloroethane
	Chlorobenzene

The average RF is calculated for all compounds. RF for both BNA and VOA SPCC compounds must be at least 0.300 except for bromoform which must be at least 0.250. The RF for all other BNA and VOA compounds must be at least 0.05.

Continuing Calibration

- Same list of CCC and SPCC for BNA and VOA analysis.
- CCC and SPCC criteria

1. % D must be less than 25% for all CCC and should be less than 25% for all other compounds for BNA and VOA analysis.
2. The RF is calculated for all compounds. RF for all other BNA and VOA SPCC compounds must be at least 0.300, except for bromoform which must be at least 0.250. The RF for all other BNA and VOA compounds must be at least 0.05.

- Continuing calibration must be performed after tune criteria is met.
- Continuing calibration must be performed before beginning sample analysis and must be done every 12 hours during analysis for BNA and VOA.

Internal Standard

Internal standard areas are monitored as a measure of the GC/MS instrument calibration. The areas of each internal standard in each sample are compared to the internal standard areas in the continuing calibration standard associated with the samples. If the samples are analyzed under the same tune as the initial or continuing calibration, the areas in each sample are compared to those in the VOA or BNA continuing calibration standard in the initial or continuing calibration.

- The area and retention time for each internal standard from the calibration standard and the upper and lower limits of the EICP area should be within -50% to +100%.
- When the retention time of any internal standard changes by more than 30 seconds, the system must be inspected and corrections made.
- The EICP area of each internal standard must fall within the limits of the 12 hour standard.

TABLE 7.1

INTERFERANT AND ANALYTE ELEMENTAL CONCENTRATIONS USED FOR ICP INTERFERENCE CHECK SAMPLE

Analytes	(mg/l)	Interferants	(mg/l)
Ag	1.0	Al	500
Ba	0.5	Ca	500
Be	0.5	Fe	200
Cd	1.0	Mg	500
Co	0.5		
Cr	0.5		
Cu	0.5		
Mn	0.5		
Ni	1.0		
Pb	1.0		
V	0.5		
Zn	1.0		

TABLE 7.2

BFB KEY IONS AND ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
50	15.0 - 40.0 percent of the base peak
75	30.0 - 60.0 percent of the base peak
95	base peak, 100 percent relative abundance
96	5.0 - 9.0 percent of base peak
173	less than 2.0 percent of mass 174
174	greater than 50.0 percent of the base peak
175	5.0 - 9.0 percent of mass 174
176	greater than 95.0 percent but less than 101.0 percent of mass 174
177	5.0 - 9.0 percent of mass 176

TABLE 7.3

DFTPP KEY IONS AND ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
51	30.0 - 60.0 percent of mass 198
68	less than 2.0 percent of mass 69
70	less than 2.0 percent of mass 69
127	40.0 - 60.0 percent of mass 198
197	less than 1.0 percent of mass 198
198	base peak, 100 percent relative abundance
199	5.0 - 9.0 percent of mass 198
275	10.0 - 30.0 percent of mass 198
365	greater than 1.00 percent of mass 198
441	present but less than mass 443
442	greater than 40.0 percent of mass 198
443	17.0 - 23.0 percent of mass 442

LABORATORY ANALYSIS

Analytical methods used by SWLO for this project include selected methods from the following: Environmental Protection Agency, (600-4-79-0020) Test Methods for the evaluation of solid waste, U.S. Environmental Protection Agency (SW 846), the U.S. EPA Methods for Chemical Analysis of Waters and Wastes. The use of other methods may be requested in writing to the laboratory manager.

Individual analytical methods are listed in section 4.0. Sample preparation methods include the following:

5030	Purged and Trap for volatile organics
3010/3020	Acid Digestion of aqueous samples for ICP/Furnace A.A. analysis
3050	Acid Digestion of soils/sediments

DATA REDUCTION, VALIDATION AND REPORTING

1. **OPERATORS:** As data comes off instruments, operators check the data to see if the data meets the QC requirements. Operators fill out QC forms. If the QC requirements are met the data then goes to the data assembly clerk. If the QC requirements are not met, then the operator takes appropriate action.

2. **DATA ASSEMBLY CLERK:** The data received from the operators is arranged according to each fraction. All the necessary forms are checked out. The clerk fills out yellow forms and sends the data to the Program Manager.

3. **DATA REVIEW SPECIALIST:** The data is reviewed for all the analytes. He makes note of any problems with the data. If corrections are required or any data is missing then the operator is informed and the missing data is generated. The operators then transfer the data to the personal computer for the diskette delivery.

4. **OPERATORS:** Operators, using the Forms Master Program, generate all the forms and the diskettes. All the data is sent to data assembly clerks.

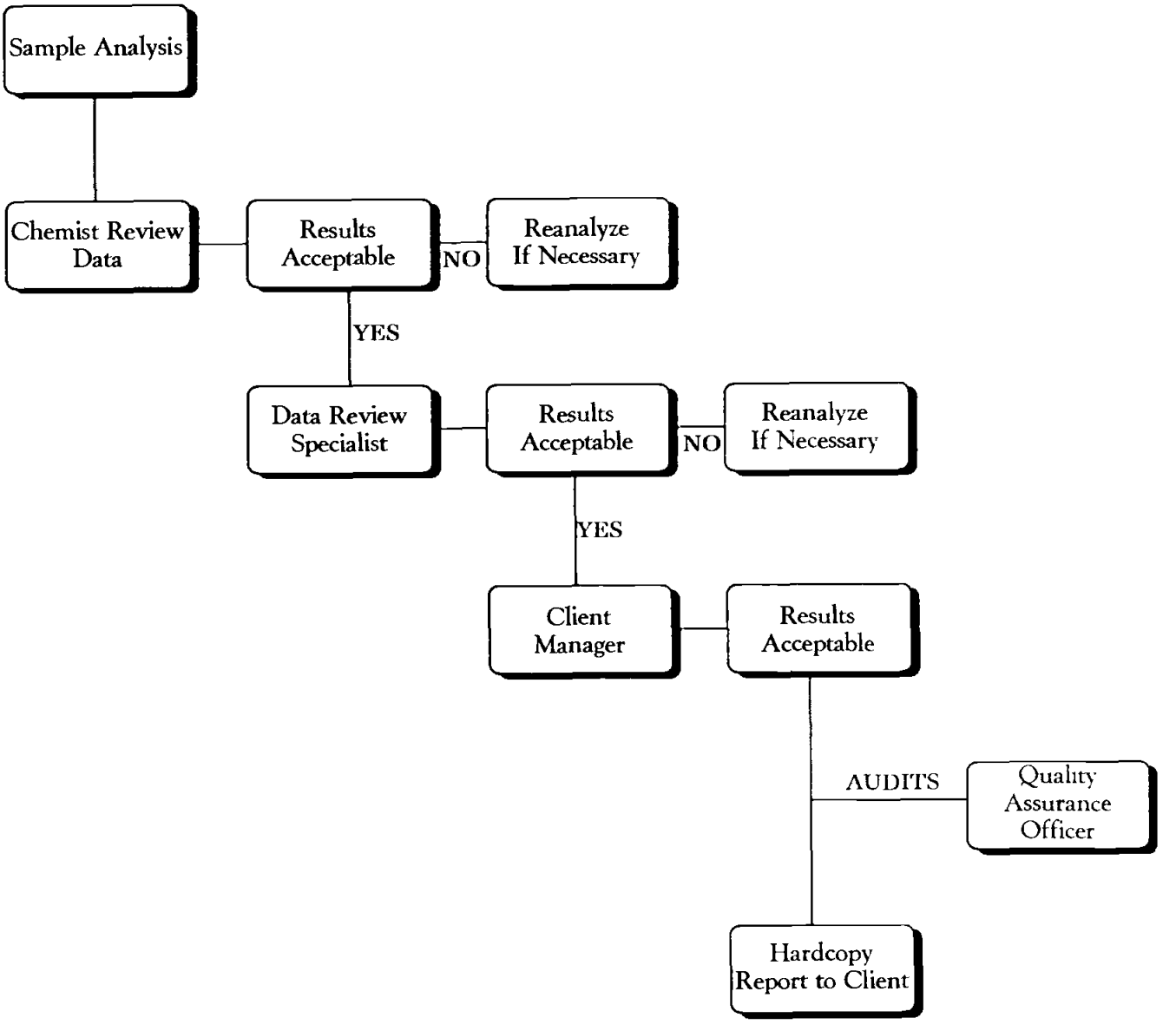
5. **DATA ASSEMBLY CLERK:** The data is arranged as per the delivery requirements. All the forms, header information and raw data are checked, the pages are numbered and the copies are made. The diskettes are labeled as per protocol.

6. **PROJECT OFFICER/PROGRAM MANAGER:** The data is reviewed again, the case narrative is written, signed and the data package is mailed. The data clerk makes note of the mailing date in a log.

7. **DATA ASSEMBLY CLERK:** The pages of the package are numbered and the appropriate number of copies are made. The copies are bound and Federal Expressed to the locations designated by the client, retaining the original in-house.

8. **QUALITY ASSURANCE OFFICER:** Review of data by the Quality Assurance Officer is routinely performed on a 10% frequency. Should the client have additional

Data Validation Scheme



INTERNAL QUALITY CONTROL CHECKS

METHOD BLANK ANALYSIS

It is the laboratory's responsibility to ensure that method interferences caused by contaminates in solvents, reagents, glassware, and other sample processing hardware be minimized.

Blanks are processed for each analysis (i.e. Volatiles, BNAs, etc.). All reagents used in the method are taken through the analytical process to ensure that contamination is minimal. Usually a blank is performed with each set of twenty (20) samples or sample matrix, whichever is more frequent. Clients are urged to send field blanks to isolate contamination introduced by field sampling techniques.

Acceptable blank criteria for each parameter is discussed in the Standard Operating Procedures.

QUALITY CONTROL

The purpose of SWLO's quality assurance plan is to ensure that the laboratory provides high-quality and cost-effective services and products to its clients. Although specific quality assurance procedures will be designed to meet the needs of each individual program, SWLO's general objectives are:

- Data should be accurate in terms of their agreement with a reference or true values.
- Data should be precise in that there is agreement among individual measurements made under similar conditions.
- Data should be complete in terms of the amount of data available vs. the amount of data evaluated.
- Data should be comparable to prior relevant data for evaluation and testing purposes.
- Data should be representative of the overall population or data base of parameter measurements.

- Data should be reproducibly obtainable under similar conditions, whether generated by the SWLO or another firm.
- Upgrade the overall quality of laboratory performance.

All of the above objectives are ensured by the QA/QC program which monitors all phases of data generation, ranging from sample collection to sample handling, to the actual analysis and data reporting that involves measurements of both inorganic and organic constituents. These procedures will be followed by all personnel and will routinely be reviewed by both the Laboratory Director and QA Officer.

Scope and Approach Relating to Measurement of Data in Terms of Precision, Accuracy, Completeness, Representativeness, and Comparability

The laboratory scope and approach to produce data of known and sufficient quality are described in this section. Guidelines are provided for the assessment and reporting of data quality for any environmentally related measurements, and for the incorporation of such assessments into major environmental data bases.

Controlled sample receiving, logging, and tracking throughout the length of the project/contract is maintained to ensure sample integrity throughout the sample analysis scheme. Documentation of instrument performance and preventive maintenance is used to provide a permanent record for data validation. SWLO routinely checks the quality of analytical work through analysis of quality control (QC) reference samples, duplicate samples, or matrix spike duplicate and spike samples.

Accuracy

The accuracy of the measurement data is evaluated by the comparison of the percent recovery of the QC reference material of known or established concentration, independent of routine calibration. It is used as prepared, or diluted with an inert matrix as a blind environmental sample. Statistically based control limits are established for each method of analysis and sample matrix. A spike sample is analyzed routinely for each batch of 20 samples (5%) and are dependent upon the sample matrix, method of analysis and concentration level. A more frequent analysis is performed (i.e., one in 10 samples) on a contract-specific basis. Recoveries are assessed to determine method efficiency and matrix interference effects. Analytical accuracy is expressed as the percent of recovery of an analyte/parameter which has been added to the environmental samples at a known concentration before preparation and analysis. The equation used to calculate percent recovery is as follows:

$$\text{Percent Recovery} = \frac{(\text{Spike Sample Result} - \text{Sample Result}) \times 100}{\text{Amount of Spike Added}}$$

Precision

The laboratory uses matrix spike duplicates to assess precision. A matrix spike duplicate is analyzed for each batch of 20 samples (5%) for in-house QC and is dependent upon the sample matrix and method of analysis. A more frequent analysis is performed (i.e., one in 10 samples) on a contract-specific basis. The basic precision statistics obtained from the multiple batch frequency may be compared to develop a graph assessment (using control limits) for given sample matrix.

Analytical precision is expressed as a percentage of the difference between the results of two matrix spike samples for a given analyte. Relative percent difference (RPD) is calculated as follows:

$$\text{RPD} = \frac{(\text{MS Result} - \text{MS Duplicate Result}) \times 100}{\text{Mean of MS and MS Duplicate Results}}$$

where MS denotes Matrix Spike.

Completeness

For the data to be valid, it must meet all the acceptance criteria including accuracy, precision and any other criteria specified by the analytical method used. Data validation procedures are employed to minimize the amount of bad data from getting through data collection.

While the quality objective is to obtain the greatest accuracy and precision, the specific accuracy/ precision level is dependent on the method of analysis and type of sample matrix. The laboratory historical statistical control limits are used as guidelines to validate the data generated unless client or contract requirements set more stringent criteria.

Representativeness

Data generated by the laboratory shall be representative of the overall population of samples collected and analyzed. It shall be representative of the laboratory data base of accuracy and precision measurements of the particular parameter(s), matrix and analytical method. If the same results are reproducible, the data can be said to represent the environmental condition.

Comparability

Data generated shall be used to evaluate completeness of extensive monitoring programs and testing purposes based on the previous data measurements of parameters, matrix and analytical method. It shall be comparable to data sets recorded in the past to check for historical consistency. In order to maximize its usefulness, data shall be reported in appropriate units and in a consistent manner. Appropriate units are in accordance with the requirements stated in the October 26, 1984, proposed rules 40 CFR, Part 136, Guidelines Establishing Test Procedures for the Analysis of Pollutants. Data should be reproducible under similar conditions whether generated by the laboratory or another firm.

Control Limits

Control limits for individual analytical procedure can be found in the section titled "QC Limits".

Instrumental Detection Limit (IDL)

It is defined as the smallest signal above background noise that an instrument can detect reliably. It does not address possible blank contaminants or matrix interferences.

IDL for each analyte in a given method is determined for each analytical instrument used. It is quarterly updated to verify instrument sensitivity changes. The following procedure is used to determine IDL:

- Using EPA or NBS supplied SRMs, if available, perform seven consecutive measurements of standards for all components being measured at 3 - 5 times the required detection limit concentrations [i.e. MDL or contract required detection limits (CQRL)] on three nonconsecutive days.
- These analyses are performed using the instrumental working conditions specified in the method, on standards in appropriate solvent for base/neutrals, acids, and pesticides/PCBs; standards diluted into reagent water for volatile organics; and trace metal analytes in reagent water.
- IDL is determined by multiplying 3 times the average of the standard deviations of the measured values.

Limit of Detection (LOD)

LOD is the lowest concentration level of an analyte that the analytical process can reliably detect. Sometimes, the IDL and LOD are operationally the same since an indication of whether an analyte signal exceeds peak-to-peak noise. LOD accounts for blank contamination but not for matrix complexity and interferences. It is also numerically equivalent to the MDL as the value

for blank approaches zero. The recommended value for LOD is 3s where s is the standard deviation of the difference between total value measured for the sample and value measured for the blank. It is expressed in an equation as:

$$S_T - S_B > 3s$$

where S_T = Total value of the analyte measured in the sample

S_B = Value of the analyte measured in the blank

s = Standard deviation for these measurements ($S_T - S_B$)

Method Detection Limit (MDL)

MDL is defined as the minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero. It also refers to the minimum concentration of an analyte that a method can detect reliably in either a given sample matrix or blank.

It is expressed in an equation as:

$$MDL = \frac{Ks}{M}$$

where K = 3
s = Standard deviation of average noise level
m = Slope of the calibration curve

Practical Quantitation Limits (PQL)

PQL is the lowest level that can be reliably achieved within the laboratory control limits of precision and accuracy during routine laboratory operating conditions. It is expressed in an equation as

PQL = [MDL x factor]

For nonaqueous samples, the factor is on a wet-weight basis.

See Appendix II for Quantitation Limits that will be used for the project.

QUALITY ASSURANCE REVIEW

Audit is defined as systematic check to determine the quality of operation of field and laboratory activities. It is comprised of the following:

- Performance audit
- System audit

Procedures used to assess the effectiveness of the quality control system are as follows:

Internal Performance Audits

These are accomplished by the laboratory through the use of control samples, replicate measurements and use of reference materials in conjunction with control charts.

Sample analysis systems are conducted by the QA Officer and include the following:

- Verification of written procedures and analyst(s) understanding
- Verification and documentation of procedures and documents
- Review of analytical data and calculations

External Performance Audits

These are accomplished by the laboratory through interlaboratory checks such as:

- Participation in laboratory evaluation programs
- Participation in round-robin method and PE studies
- Participation in PE samples available from EPA
- Analysis of split samples and comparing results with the other laboratory

Laboratory System Audit

An on-site inspection is done by EPA audit team or laboratory certification personnel to review the laboratory quality control system which covers sample handling, sample analysis, records control, preventive maintenance, and proficiency testing. When EPA or laboratory certification personnel initiate a system audit of the laboratory, any recommendations made or deficiencies identified will be considered for implementation and corrective actions taken to correct deficiencies.

PREVENTATIVE MAINTENANCE

Southwest Laboratory has developed an in depth program for preventative maintenance. Simplified operations are maintained by the chemist and technicians. The more difficult and intricate maintenance operations are scheduled and organized by the "Organic or Inorganic Program Manager".

Preventative maintenance schedules are placed in the front of each individual instrument logbook. Information acquired in this schedule would be written as the maintenance is performed. Maintenance to be performed is scheduled for the Organic or Inorganic Programs Manager's officer.

Table 12.1 summarizes the preventative maintenance requirements for Southwest Laboratory. Instrument identification, items to be checked or serviced and the frequency in which each operation is performed are listed.

TABLE 12.1

Preventative Maintenance Requirement
Southwest Laboratory Operations

INSTRUMENT	ITEMS CHECKED/SERVICED	FREQUENCY
Gas Chromatography	Replace column packing, clean detector, Change glass wool plug, Clean insert, Replace septa, Gas purity checks.	Determined by analyst so that the calibration is within the specifications.
Atomic Absorption Spectrometer	3 point calibration performed, burner head, Nebulizer, Tygon Tubing	Daily Daily Monthly 6 months
Inductively Coupled Plasma (ICP)	Capillary and pump tubing Liquid Argon Tank Slit Micrometer (reprofile) Replace/Realign Plasma Torch Nebulizer Primary Imaging Mirror Photomultiplier Alignment	Twice weekly Weekly Daily As needed Monthly Weekly As needed
Ion Chromatograph	Check plumbing, Check filter (inlet) Flush column check bed support	Daily Weekly After each sample
GC/MS	GC/MS maintenance is the same as GC with the following additions:	
	DP Oil Mech Oil Power Con. Air Filter QEM Filter Water Filter Vacuum Chaff Filter Computer Air Filter Card Gage Air Filter Interface Box	Bi-weekly Quarterly Bi-weekly Bi-weekly Bi-weekly Monthly Bi-monthly Monthly Bi-weekly

SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA PRECISION ACCURACY & COMPLETENESS

See QA Objectives (Section 4.0), Internal Quality Control Checks (Section 10.0)

CORRECTIVE ACTIONS

When errors, deficiencies or out-of-control situations exist, the QA plan provides systematic procedures called "corrective actions" to resolve problems and restore proper functioning of the analytical system. Laboratory personnel are alerted that corrective actions may be necessary if:

1. Data is outside the warning or acceptable windows for precision and accuracy.

2. Undesirable trends in concentration, spike recoveries and relative percent difference (RPD) are detected.

3. There are unusual changes in detection limits.

4. Deficiencies are detected by the QA Officer during internal and external walk-throughs, or from the results of performance evaluation samples.

5. Complaints are received from the clients.

Corrective action procedures are often handled at the bench level by the analyst, who will review the extraction procedure for possible errors, check the instrument calibration, spike mixes and standard mixes, instrument sensitivity, etc. If the problem persists or cannot be identified, the matter is referred to the laboratory supervisor, manager and/or QA Officer who will further investigate. When the problem is resolved, the QA Officer is provided with full documentation, which is kept on file in the QA office.

QUALITY ASSURANCE REPORTS TO MANAGEMENT

See Responsibilities (Section 3.0)

SOUTHWEST LABORATORY OF OKLAHOMA, INC.

STANDARD OPERATING PROCEDURE FOR THE ANALYSIS OF METALS

MT910

REV 1.0 —4/16/91

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INDUCTIVELY COUPLED ARGON PLASMA

I. INTRODUCTION

This method is used for the determination of dissolved, suspended or total metals in a variety of matrices. Table I lists elements for which this method applies with current wavelengths used and laboratory detection limits. Elements and wavelengths will be added as new methods are developed. Detection limits are updated quarterly.

This method describes the technique for the simultaneous multi-element determination of trace metals in solution using a Thermal-Jarrel Ash ICAP-61. The basis of this method is the measurement of atomic emission by an optical spectroscopic technique. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where excitation occurs. Characteristic atomic-line emission spectra are produced by a radio-frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer and the intensities of the lines are monitored by photomultiplier tubes. The photo currents from the photomultiplier tubes are processed and controlled by a computer system.

Table I. Elements Analyzed by ICP and their corresponding wavelengths and detection limits.

Element	Wavelength (nm)	Detection Limits, ug/L
Aluminum	308.215	36.0
Antimony	217.581	25.8
Arsenic	193.606	32.1
Barium	493.409	16.1
Beryllium	313.042	0.7
Boron	249.678	42.0
Cadmium	228.802	2.8
Calcium	317.933	207.0
Chromium	267.716	1.9
Cobalt	228.616	7.6
Copper	324.754	3.9
Iron	249.940	27.0
Lead	220.353	17.4
Magnesium	279.079	126.0

continued...

continued.....

Table I. Elements Analyzed by ICP and their corresponding wavelengths and detection limits.

Element	Wavelength (nm)	Detection Limits, ug/L
Manganese	257.610	2.5
Mercury	194.220	50.0
Molybdenum	202.030	32.9
Nickel	221.604	6.2
Osmium	225.585	—
Phosphorus	214.914	100.0
Potassium	766.491	453.0
Selenium	196.026	27.7
Silicon	288.158	100.0
Silver	328.068	9.0
Sodium	588.995	287.0
Thallium	190.864	32.4
Tin	189.989	80.0
Titanium	334.941	80.0
Vanadium	292.402	6.0
Zinc	213.856	4.0

II. INTERFERENCES

A. Spectral Interferences

1. Overlap of a spectral line from another element compensated by interelement correction by instrument.
2. Unresolved overlap of molecular band spectra may require selection of an alternate wavelength.
3. Background contribution from continuous or recombination phenomena.
4. Background contribution from stray light from the line emission of high concentration elements.

Note: Number 3 and 4 effects can usually be compensated by a background correction adjacent to the analyte line.

B. Physical Interferences - affects associated with the sample nebulization and transport processes. Dilution by be necessary.

III. APPARATUS

A. Thermal-Jarrel Ash ICAP-61 optical emission spectrophotometer.
B. IBM PC/AT computer with ThermoSPEC operating software. Automated background correction and interelement corrections.
C. Radio frequency power supply with 2500 watts capacity.
D. Liquid argon supply, 4300 cu.ft.
E. Gilson - 240 Autosampler with 240 sample positions and 14 QA/QC position.

IV. OPERATING CONDITIONS

A. Start-Up

1. Check that the argon tank main valve is open and the pressure is set to at least 60 psi.
2. Turn on the plasma work coil coolant water and exhaust vents.
3. Check that the drain tube is inserted into a plastic water bottle containing at least 8 inches of water.
4. On the R.F. generator control panel, turn the forward power manual control rheostat knob counter clockwise to the off position.
5. Turn the line and control circuit breaker switches on. The white power on lamp should be illuminated.
6. Turn the torch gas toggle switch on and adjust flow meter to 18, turn the auxiliary flow on to 0.5 and the sample gas flow to 0.6.
7. Purge the torch surfaces, the capillary tube and the drainage tube of air for at least 3 minutes.
8. Check that the blue R.F. off lamp on the generator is illuminated.
9. Turn the Automatic Power Control switch to the manual position.
10. Press the red R.F. on button.
11. Turn the sample gas flow control knob counter clockwise until the flowmeter ball stops falling.
12. To ignite, slowly turn the forward power manual control rheostat knob clockwise until the forward power meter reads 0.5 KW.
13. Press the ignitor button on the front panel of the generator. You should see a faint filamentary plasma swirling in the outer tube of the torch. Gradually increase the forward power until the plasma ignites.
14. Once the plasma is lit, rotate the forward power rheostat knob until the forward power meter reading is 1.0 - 1.1 KW.
15. Turn the automatic power control switch to the automatic position.
16. Introduce the rinse solution into the plasma by rotating the sample gas control knob to a reading of 0.6.

B. Shutdown

1. Press the R.F. OFF button on the R.F. generator control panel.
2. Turn off the gas at the tank while the toggle switches are still open. When both gauges on the tank read 0, turn OFF the toggle switches.
3. Turn off the torch coolant water.
4. Turn the R.F. generator line and control circuit breakers off.

V. REAGENTS AND STANDARDS

A. Nitric Acid, conc.: Baker Instra-analyzed for trace metal Analysis.
B. Nitric Acid (1:1): Add 500 ml conc. HNO_3 to 400 ml ASTM Type II water and dilute to 1 liter.
C. Hydrochloric Acid, conc.: Baker Instra-analyzed for trace metal analysis.
D. Hydrochloric Acid (1:1): Add 500 ml conc HCl To 400ml ASTM Type II water and dilute to 1 liter.
E. Hydrogen Peroxide, 30%: Method Control Blank meets ACS Standards.
F. Standard Stock Solutions

1. Inorganic Ventures, Inc. - use high purity inorganics as starting materials. Place into solution with purified acids and 18 mega-ohm double deionized water. All standards are traceable to national standards.
2. Spex Industries, Inc. - prepared from 99.995% - 99.999% pure elements assayed by wet chemical methods dissolved in 18 mega-ohm, double deionized water and electronic grade acids. Date solutions are received and opened are posted on each bottle.

G. Blank Solutions

1. Calibration blanks - dilute 2.0 ml of (1:1) HNO_3 and 10.0 ml of (1:1) HCL to 100 ml with ASTM Type II water. This blank is used in establishing the analytical curve and to flush the system between standards and samples.
2. Preparation Blank - contains all the reagents and in the same volumes as used in the processing of the samples. The preparation blank is carried through the complete digestion procedures and contains the same acid concentration in the final solution as the sample solution.

H. Calibration Check Standard: A standard made from a different stock solution as that used for the calibration standards and analyzed

immediately after calibration to validate calibration. Concentration of the calibration check standard is approximately at the mid-point of the calibration curve.

VI. SAMPLE PREPARATION

A. Separate operating procedures are written for the digestion of samples to be analyzed by the ICP depending on the matrix of the sample.

VII. ANALYSIS

A. Set up the instrument with proper operating parameters already established. The instrument must be allowed to become thermally stable before beginning. This usually requires at least 30 minutes of operation prior to calibration.
B. Initiate appropriate method file configuration on computer.
C. Calibrate the ICP starting with the highest mixed standard. Flush the system with the calibration blank between each standard. Use at least three replicate exposures for both standardization and sample analysis.
D. Begin the sample run by analyzing the calibration blank and calibration verification check samples. Analyze the blank and calibration check samples every 10 samples. Flush the system with the calibration blank between each sample.
E. Record the analytical sequence on the ICPrun log.

VIII. CALCULATION

A. If dilutions were performed, the appropriate factor must be applied to sample values.
B. Data should be reported in ug/L up to three significant figures.
C. When rounding off values, use guidelines specified in EPA "Handbook for Analytical Quality Control in Water and Wastewater Laboratories".

IX. QUALITY CONTROL

A. Initial Calibration Verification

1. Immediately after the ICP has been calibrated, the accuracy of the initial calibration is verified and documented for every analyte by the analysis of EPA Initial Calibration Verification Solution at each wavelength used for analysis. If the measurements exceed the control limits of 90 - 110% of the true value, the analysis must be terminated, the problem corrected, the instrument recalibrated. and the calibration reverified.

ICP RUN LOG

ANALYST: ______________________ DATE ______________________

INSTRUMENT: ______________________ CALIBRATION STD: SOURCE ______________________

Client ID	SWLO ID	Time	Percent Recovery	Dilution Factor	Analytes (circle)
					Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, V, Zn
					Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, V, Zn
					Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, V, Zn
					Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, V, Zn
					Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, V, Zn
					Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, V, Zn
					Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, V, Zn
					Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, V, Zn
					Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, V, Zn
					Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, V, Zn
					Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, V, Zn
					Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, V, Zn
					Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, V, Zn
					Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, V, Zn
					Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, V, Zn
					Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, V, Zn
					Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, V, Zn
					Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, V, Zn
					Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Sb, V, Zn
					Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn Na Ni, Pb, Sb, V, Zn

B. Continuing Calibration Verification

1. To ensure calibration verification during the analysis run, a continuing calibration verification standard is analyzed for every analyte at each wavelength at a frequency of 10% or every 2 hours during an analysis run, whichever is more frequent. The standard is also analyzed for every wavelength used for analysis at the beginning of the run and after the last analytical sample. The same continuing calibration standard must be used throughout the analysis runs for a case of samples received. If the measurements exceed the control limits of 90-110% of the true value, the instrument must be recalibrated and the preceding samples analyzed since the last good calibration verification must be reanalyzed.

C. CRDL Standard

1. To verify linearity near the CRDL an ICP standard at two times the CRDL or two times IDL, whichever is greatest, is analyzed at the beginning and end of each sample analysis, or a minimum of twice per 8 hour working shift, whichever is more frequent. This standard is run for every wavelength used for analysis except these for Aluminum, Barium, Calcium, Iron, Magnesium, Sodium, Potassium. No control limits for this standard have been set at this time.

D. Initial and Continuing Calibration Blank

1. A calibration blank is analyzed at each wavelength used for analysis immediately after each every initial and continuing calibration verification, at a frequency of 10% or every 2 hours during the run, whichever is more frequent. If the absolute value of the blank exceeds the CRDL, terminate analysis, correct the problem, recalibrate and reanalyze all analytical samples analyzed since the last good calibration blank.

E. Preparation Blank

1. At least one preparation blank must be prepared and analyzed with every Sample Delivery Group (SDG), or with each batch of samples digested, whichever is more frequent. This blank is used to ascertain whether sample concentrations reflect contamination in the following manner:

a. If the absolute value of the blank is less than or equal to the CRDL no corrective action is necessary.

b. If any analyte concentration in the blank is above the CRDL, the lowest concentration of that analyte in the associated samples must be 10x the blank concentration. If not, all samples less than 10x the blank concentration and above the CRDL, must be redigested and reanalyzed for that analyte.

c. If the concentration of the blank is below 10x CRDL, all samples associated with the blank must be redigested and reanalyzed.

F. Interference Check Sample (ICS)

1. To verify interelement and background correction factors an ICP interference check sample is analyzed at the beginning and end of each analysis run or a minimum of twice per 8 hour working shift, whichever is more frequent. The ICS is obtained from EPA and consists of two solutions. Solution A consists of the interferants and solution AB consists of the analytes mixed with the interferants. An ICS analysis consists of analyzing both solutions consecutively for all wavelengths used. Results for the analyses of solution AB must fall within the control limits of $\pm$ 20% of the true value for the analytes. If not, terminate the analysis, correct the problem, recalibrate and reanalyze the analytical samples analyzed since the last good ICS.

G. Matrix Spike Sample

1. The spike solution is added before digestion to provide information about the effect of the sample matrix on the digestion and measurement methodology. At least one spike analysis is performed on each group of samples of a similar matrix type and concentration or for each Sample Delivery Group (SDG), whichever is more frequent.
2. Samples identified as field blanks cannot be used for spiked sample analysis.
3. If the spike recovery is not within the limits of 75-125% the data of all samples received associated with that spike sample and determined by the same analytical method will be flagged with the letter "N". An exception to this is when the sample concentration exceeds the spike concentration by a factor of four or more.

H. Post Digested Spike

1. When the pre-digested spike recovery falls outside the control limits and the sample result does not exceed 4x the spike added, a post-digested spike is performed except for Silver.
2. Spike the unspiked aliquot of the sample at 2x the indigenous level or 2x CRDL whichever is greater.

I. Duplicate Sample

1. One duplicate sample is analyzed from each group of samples of a similar matrix type and concentration or for each Sample Delivery Group, whichever is more frequent.
2. Samples identified as field blanks cannot be used for duplicate sample analysis.
3. A control limit of 20% for relative percent difference (RPD) is used for original and duplicate sample values greater than or equal to 5x CRDL. A control limit of $\pm$ CRDL is used for sample values less than 5x CRDL.

J. Laboratory Control Sample (LCS)

1. Aqueous and solid LCS are analyzed for each analyte using the same sample preparations, analytical methods and QA/QC procedures employed for the samples. The aqueous LCS is obtained form EPA (or use the initial calibration verification solution). One aqueous LCS is prepared and analyzed for every group of aqueous samples in a Samples Delivery Group, or for each batch of aqueous samples digested, whichever is more frequent. If the percent recovery for the aqueous LCS falls outside the control limits of 80-120% (exception: Ag and Sb), the analyses must be terminated, the problem corrected, and the samples associated with that LCS redigested and reanalyzed.
2. The EPA provided solid LCS is prepared and analyzed using the same procedures as the samples, one solid LCS must be prepared and analyzed for every group of solid samples in a Sample Delivery Group, or for each batch of samples digested, whichever is more frequent. If the result for the solid LCS falls outside the control limits established by the EPA, the analyses must be terminated, the problem corrected and samples associated with that LCS redigested and reanalyzed.

K. ICP Serial Dilution

1. The ICP Serial Dilution analysis is performed on each group of

samples of a similar matrix type and concentration or for each Sample Delivery Group, whichever is more frequent.

2. Samples identified as field blanks cannot be used for serial dilution analysis.
3. If the analyte concentration is at least a factor of 50 above the instrument detection limit in the original sample, an analysis of a 5 fold dilution must agree within 10% of the original determination.

L. Instrument Detection Limits

1. Detection limits for the ICP are determined quarterly (every 3 months). The detection limits are determined on three nonconsecutive days using the following protocol:

   a. A low level standard at three to five times the estimated IDL is measured seven times and the standard deviation is multiplied by three.

   b. The average detection limit for the three days is the IDL.

M. Interelement Corrections

1. Interelement corrections are determined quarterly by analyzing 1000 ppm standards of the interferants and determining the effect on the analytes.

N. Linear Ranger

1. A linear range check standard is analyzed quarterly for each element analyzed by ICP. The analytically determined concentration must be within $\pm$ 5% of the true value. This concentration is the upper limit of the ICP linear range beyond which results cannot be reported without dilution of the analytical sample.

MATRIX SPIKE SOLUTIONS

SUMMARY

At least one predigested spike sample analysis must be performed on each group of samples of a similar matrix type and concentration or for each Sample Delivery Group, whichever is more frequent. The following is the procedures for preparing the spiking solutions used.

ICP/AA SOLUTION #1

Using Class A volumetric pipettes, add IV-30, IV-40, 1000 ppm antimony and cadmium at the following volumes to a 100 ml volumetric flask. Add 2 ml of conc. HNO_3 and dilute to 100 ml with ASTM Type II water.

ELEMENT	Conc. of Stock Sol.	ml of stock Sol./100 ml	Final Conc. spiking sol.(ppm)	Conc. in matrix spike (ug/L)
Aluminum	20000	1.0	200	2000
Antimony	1000	5.0	50	500
Barium	20000	1.0	200	2000
Beryllium	50	10.0	5	50
Cadmium	1000	0.5	5	50
Chromium	200	1.0	20	200
Cobalt	500	10.0	50	500
Copper	250	10.0	25	250
Iron	1000	10.0	100	1000
Manganese	500	10.0	50	500
Nickel	500	10.0	50	500
Vanadium	500	10.0	50	500
Zinc	500	10.0	50	500

For aqueous samples add 1.0 ml of spiking solution. Final volume of digestate = 100 ml.

For soil samples add 2.0 ml of spiking solution. Final volume of digestate = 200 ml.

ICP/AA SOLUTION #2

Using a Class A volumetric pipette add the 1000 ppm stock standard at the following volume to a 100 ml volumetric flask. Add 2.0 ml of conc. HNO_3 and dilute to 100 ml with ASTM Type II water. This solution should be made fresh weekly.

ELEMENT	ml of 1000 PPM Std/100 ml	Conc. of Spiking Solution (PPM)	Final Conc. in Matrix Spike (ug/L)
Silver	0.5	5	50

For aqueous samples add 1.0 ml of spiking solution. Final volume of digestate = 100 ml.

For soil samples add 2.0 ml of spiking solution. Final volume of digestate = 200 ml.

ICP/AA SOLUTION #3

Using Class A volumetric pipettes add the 100 ppm stock standards at the following volumes to a 100 ml volumetric flask. Add 2 ml of conc. HCl and dilute to 100 ml with ASTM Type II water.

ELEMENT	ml of 1000 PPM Std/100 ml	Conc. of Spiking Solution (PPM)	Final Conc. in Matrix Spike (ug/L)
Arsenic	20.0	200	2000
Lead	5.0	50	500
Selenium	20.0	200	2000
Thallium	20.0	200	2000

For aqueous samples add 1.0 ml of spiking solution. Final volume of digestate = 100 ml.

For soil samples add 2.0 ml of spiking solution. Final volume of digestate = 200 ml.

FURNACE SOLUTION #4

Using Class A volumetric pipettes make a 100 ppm intermediate stock solution for the elements listed below. This is done by adding 10 ml of the

1000 ppm stock solution to a separate 100 ml volumetric flask. Add 2 ml of conc. HNO_3 and dilute to 100 ml. For the matrix spike solution add the appropriate amounts of the 100 ppm intermediate stock solution to a 100 ml volumetric flask. Add 2 ml of conc. HNO_3 and dilute to 100 ml with ASTM Type II water.

ELEMENT	ml of 1000 PPM Std/100 ml	Conc. of Spiking Solution (PPM)	Final Conc. in Matrix Spike (ug/L)
Arsenic	4.0	4.0	40
Cadmium	0.5	0.5	5
Lead	2.0	2.0	20
Selenium	1.0	1.0	10
Thallium	5.0	5.0	50

For aqueous samples add 1.0 ml of spiking solution. Final volume of digestate = 100 ml.

For soil samples add 2.0 ml of spiking solution. Final volume of digestate = 200 ml.

MERCURY SOLUTION #5

Using Class A volumetric pipettes make a 10 ppm intermediate stock solution by adding 1.0 ml of the 1000 ppm stock solution to a 100 ml volumetric flask. Add 2 ml of conc. HNO_3 and dilute to 100 ml. For the matrix spike solution add the appropriate amount of the 10 ppm intermediate stock solution to a 100 ml volumetric flask. Add 2.0 ml conc. HNO_3 and dilute to 100 ml with ASTM Type II water.

ELEMENT	ml of 1000 PPM Std/100 ml	Conc. of Spiking Solution (PPM)	Final Conc. in Matrix Spike (ug/L)
Mercury	1.0	100	1.0

For both aqueous and soil samples add 1.0 ml of spiking solution. Final volume of digestate = 100 ml.

The matrix spike solution should be made fresh each day of use.

CYANIDE SOLUTION #6

Using a Class A volumetric pipette add the 1000 ppm stock standard at the following volume to a 100 ml volumetric flask. Add 1.0 gram of NaOH and dilute to 100 ml with ASTM Type II water. This solution should be made fresh daily.

ELEMENT	ml of 1000 PPM Std/100 ml	Conc. of Spiking Solution (PPM)	Final Conc. in Matrix Spike (ug/L)
Cyanide	5.0	50	100

For both aqueous and soil samples add 1.0 ml of spiking solution. Final volume of digestate = 250 ml.

SUMMARY OF FURNACE ANALYSIS

I. Samples are received from clients

II. Sample log in to LIMS system

A. Sample Custodians will assign the proper test codes to each sample according to the customers traffic log.

III. Sample Preparation for Furnace and Mercury Metals

A. By looking at LIMS the Inorganic or Metals Program Manager will assign digestions that are required.

B. The Sample Prep Technician will then digest or prep samples according to the procedure of the methods used.

C. After being prepped, he/she will place the digestion in the designated cabinet.

IV. Worksheets and Priority List

A. The Furnace Supervisor or the Senior Furnace Operator will make up the worksheets for the furnace metals.

B. Then they will place the digestion on the priority list.

V. Analysis for Required Metals

A. The first thing the analyst will decide is which digestion he/she will do. This will be dictated by the priority list.

B. Before starting the actual analysis, the analyst will decide what protocol they will have to use (either Private or CLP), then run samples according to that protocol.

C. Once an analyst finishes what he/she is working on, they will look the priority list and the folder with the rest of the worksheets, and start working on the next group of digested samples that is due.

VI. Write-ups for Each Run to be Done

A. When the run is done, the analyst will fill out the worksheet and do

the write-up according to protocol.

VII. Data Review

A. Once the write-up is done it will be required to be given to the Furnace Supervisor or the Senior Furnace Operator for data review.

VIII. Data Given to Data Clerks

A. After the data has been reviewed, all CLP protocol data will go to the Inorganic Data Clerks, and the Private protocol will go into the instrument box for storage.

IX. Data Taken off LIMS and Workbook

A. After the data has been reviewed and the digestion is completely analyzed, the Furnace Supervisor will designate a person to enter the results in LIMS and put it in the book if necessary.

X. Storage and Disposal of Samples

A. After each digestion has been analyzed for all elements required, they will be stored for a predetermined length of time.

B. After the predetermined length of time is up, the samples will be disposed of according to protocol.

PREPPED FURNACE CASES

01/28

SDG #	DUE DATE	As	Pb	Se	Tl	BOX #	# OF BILLABLE	DATE DONE
4838.01 /	01\31	XXX	XXX	XXX	XXX	4,7	15	01\21
4796 01 /	02\02	XXX	XXX	XXX	XXX	2	1	01\21
4824.01 /	01\24	XXX	XXX	XXX	XXX	26	1	01\21
4837 01 /	01\24	XXX	XXX	XXX	XXX	27	6	01\21
4833.01 /	01\24	XXX	XXX	XXX	XXX	26	7	01\22
4852.01 / MX2943	02\19	000	000	000	000	12	5	
4852.03 / MX2945	02\19	000	000	000	000	21	4	
4850.01 / MFN401	02\18	XXX	000	XXX	XXX	16	11	
4850.03 / MFN403	02\18	000	000	000	000	14	14	
4867 01 / B&MC	02\12	XXX	000	000	XXX	1	3	
4857 01 /	02\24	XXX	XXX	000	XXX	11	7	
4868.01 / MDY663	02\21	000	===	000	===	25	15	
4882 01 / PORTCOE	02\06	XXX	000	XXX	XXX	5,6,8	26	

WORKSHEET

MT

WATERS BOX 10

EPA CLP

#-	SAMPLE# \ CLIENT ID #	DATE DUE	DATE\ ANALST	COMMENTS	RE- SULT
01-	PBW \ PBW	02\07\91			
02-	LCSW \ LCSW	02\07\91			
03-	4751.01 \ MFK914	02\07\91			
04-	4751.01D \ MFK914D	02\07\91			
05-	4751.01S \ MFK914S	02\07\91			
06-	4751.02 \ MFK915	02\07\91			
07-	4767.01 \ MFK916	02\07\91			
08-	4767.02 \ MFK917	02\07\91			
09-	4781.01 \ MFK909	02\07\91			
10-	4781.02 \ MFK910	02\07\91			
11-	4792.01 \ MFK912	02\07\91			
12-	4792.02 \ MFK913	02\07\91			
13-	4792.03 \ MFK918	02\07\91			
14-	\				
15-	\				
16-	\				
17-	\				
18-	\				
19-	\				
20-	\				

GRAPHITE FURNACE ATOMIC ABSORPTION SPECTROMETRY

I. Instrumentation

Here at AATS, we have the following four furnace instruments:

A. 1 - Perkin Elmer 5000 spectrophotometer HGA-500 Furnace Zeeman background correction
AS-40 auto sampler
As, Se EDL lamps

B. 1 -Thermo Jarrell Ash Video 22E AA spectrophotometer
CTF 188 Atomizer
Smith-Hieftye background correction
ISC 75 microprocessor controlled auto sampler
Pb, Tl HCL lamps

* The TJA22 is also supplied with air which powers the door mechanism on the auto sampler.

C. 2 -Perkin Elmer 5100 spectrophotometer
HGA-600 Furnace
Zeeman background correction
AS-60/70 auto sampler
As, Pb, Se, Tl EDL lamps
Pb, Tl HCL lamps

* All instruments are supplied with zero grade Argon and cooled with water coolers filled with distilled water.

II. Daily Operation

A. If the analyst has any problem with the daily operation of the instruments, they are instructed to look at the accompanying manuals

III. Theory of Graphite Furnace Atomic Absorption Spectrophotometry

A. Every analyst should thoroughly understand the principles and practices of atomic absorption. Should you need references upon this

subject, consult the following text:

Graphite Furnace AAS, a Source Book
Slavin, W.

Concepts, Instrumentation, and Techniques
in Atomic Absorption Spectrophotometry
Beaty, R.D.

IV. Safety Information

A. Never look directly at the furnace during the atomization state without the proper eye protection.

B. Never touch the atomizer cell until it has returned to ambient temperature.

C. Follow standard safety procedures for the handling of hazardous materials.

V. Parameters

A. Parameters for each instrument and element are placed in a logbook (which is explained in the last section of this SOP). It should be noted that the temperatures are employed as guidelines. Since temperature sensing mechanisms and temperature controllers can vary with time, the validity of the furnace parameters must be periodically confirmed by systemically altering the furnace parameters while analyzing a standard. In this manner, losses of analyte due to higher than necessary temperature settings or losses in sensitivity due to less than optimum settings can be minimized.

VI. Routine Maintenance

A. Everyday before replacing the graphite tube, wipe off the excess carbon residue on the contact rings.

B. As the contact rings are being wiped off, check for wear and if worn, replace them.

C. Clean the quartz windows daily with methanol and dry with a soft, lint-free cloth.

D. Clean the workhead surface daily.

E. Make sure the water reservoir is full and empty the waste bottle daily.

F. For other required maintenance, check with the accompanying instrument manual.

* Directions for changing contact rings and replacing graphite tubes are in the respective instrument manuals.

VII. Trouble Shooting

A. Each instrument manual has a section on trouble shooting common problems within each system. It is therefore advised that each analyst consult these manuals first.

B. If the instrument manual fails to show any light on the problem, it is advised to ask one of the managers or the Lead Furnace Operator.

C. If none of these are around, you are advised to call one of the following:

Electronics Plus (Alan Brasil or Rollie Scot)
Telephone Number (918) 250-1505
Electronics Plus has our maintenance contracts for the TJA22 and the PE5000. They will be helpful for most problems with these instruments.

Perkin-Elmer (Ernie Riggs or Art Ingraham)
Telephone Number (918) 743-5302 or 743-5303
Ernie and Art are our Field Service Engineers for both of the PE5100s. Call either one of them if we have any mechanical problems with the instrument. They will also be able to answer a limited amount of technical questions.

Perkin-Elmer (Martha Cole)
Telephone Number (713) 530-5554
Martha is an applications specialist and should be able to answer all technical questions.

Thermo-Jarrel Ash (ask for AA specialist)
Telephone Number (508) 520-1880
At this phone number you should be able to get any answers you have that Electronics Plus could not answer.

ELEMENTAL ANALYSIS — AS, PB, SE, TL

I. APPLICATION

A. Tested Concentration Range:

1. Arsenic: 10.0 - 100.0 ug/l

2. Lead: 3.0 - 100.0 ug/l (Perkin-Elmer 5100s)
   3.0 - 50.0 ug/l (TJA22)

3. Selenium:5.0 - 50.0 ug/l

4. Thallium: 10.0 - 50.0 ug/l

B. Instrument Detection Limits

We will determine these every three months as required by the EPA. They are determined by running 7 consecutive measurements of a 3x - 5x standard solution on three nonconsecutive days. Then taking the standard deviations of each day and adding them together, we will receive the IDL for that instrument.

C. Interferences

1. Molecular absorption bands - the use of background correction should eliminate this interference.

2. Memory effects - clean tube by operating at atomization temperature.

3. Carbide formation - the use of a pyrolytically coated graphite tube reduces this interference.

4. Anion interference - the use of nickel nitrate matrix modifier reduces this effect.

D. Safety Information

1. Never look directly at the furnace during the atomization state without the proper eye protection.

2. Never touch the atomizer cell until it has returned to ambient temperatures.

3. Follow standard safety procedures for the handling of hazardous materials.

II. PREPARATION OF STANDARDS

A. Source of all the standards we use are from separate Plasma-Chem standards containing 1000.0 mg/l of each of the elements. The exception is the standard for the ICV and LCSW, which we receive from the EPA.

B. Two intermediate standards are made every 8 weeks, while two more intermediate standards are made every week.

C. All calibration standards are made up every day.

D. All calibration standards are made up with ASTM Type II water and made as 1% HNO . Specific directions of how to make them up and when, are at the beginning of the calibration and standards notebook.

III. INSTRUMENT CALIBRATION

A. Calibration

1. Set up spectrophotometer, furnace and autosampler according to manufacturers recommendations.

2. Analyze the calibration blank to establish the baseline.

3. Optimize the instrument by adjusting operating parameters to achieve maximum response.

4. Beginning with the blank and working toward the highest standard, inject the solutions and record the readings.

B. Analysis of Calibration Data

1. Tabulate the calibration standard concentration versus peak area response for each calibration standard.

2. Analyze the data by linear regression on calculator. Correction coefficients must be greater than 0.995 before proceeding with analysis.

IV. INSTRUMENTAL ANALYSIS

A. Set up instrument parameters according to current optimized parameters, as discussed in Section V of the Graphite Furnace AA Spectrometry.

B. Calibrate instrument following procedure in Section III.

C. Analyze samples using the appropriate Protocol as explained in the next section.

PROTOCOL — CLP & PRIVATE

I. CALIBRATION CHECKS

A. Initial Calibration Verification

1. After the system has been calibrated, the accuracy of the initial calibration shall be verified and documented by the analysis of an EPA supplied standard at a concentration other than that used for calibration, but within the calibration range.

2. The measurement must be within + or - 10% of the trace value or the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration reverified.

B. Calibration Blank

1. A calibration blank is analyzed immediately after the calibration verifications.

2. Blanks are to be reported down to the instrument detection limit.

3. If the absolute value of the blank is greater than 3.0 ppb, terminate analysis, correct the problem and recalibrate.

C. Continuing Calibration Verification

1. To assure calibration accuracy during each analysis run, an independently prepared standard solution, will be analyzed at the frequency of 10% or 20 injections after the last analytical sample.

2. If the deviation of continuing calibration verification is greater than + or - 10% of the true value, the instrument will be recalibrated and the proceeding 10 samples reanalyzed.

D. Sample Analysis

1. Analyze each sample, except during Full Method of Standard Addition (MSA) with duplicated injections. If the value is within the calibration range, record the concentration values for both injections, the average value and the coefficient of variation (CV). For concentrations greater than the CRDL, the CV between duplicate injections must agree within 20%, or the sample must be rerun once.

2. All analytical samples, except the predigested spike sample, requires an analytical spike to determine if the MSA will be required for quantitation. For lead the spike concentration will be 20.0 ppb., all others will be 2 x CRDL. The analytical spike of a sample will be run immediately after that sample. The percent recovery (%R) of the spike will then determine how the sample will be quantitated as follows:

   a. If the spike recovery is less than 40%, the sample will be diluted by a factor of 5 and rerun with another spike. If after dilution the spike recovery is still <40%, the data will be reported and flagged with an "E" to indicate interference problems.

   b. If the spike recovery is greater than 40% and the sample concentration is <50% of the spike, the sample result will be reported to the IDL. If the spike recovery is less than 85% or greater than 115%, the data will be flagged with a "W".

   c. If the sample concentration is >50% of the spike and the spike recovery is between 85%-115%, the sample will be quantitated directly from the calibration curve and reported to the IDL.

   d. If the sample concentration is $\geq$ 50% of the spike and the spike recovery is <85% or >115%, the sample must be quantitated by MSA.

3. The following will be used for MSA analysis:

   a. Data from MSA calculations will be within the linear range as determined by the calibration curve generated at the beginning of the analytical run.

   b. The sample and three spikes will be analyzed consecutively for MSA quantitation, only single injections will be used.

   c. Spikes will be prepared such that:

      Spike 1 - Is approximately 50% of the sample absorbance.

      Spike 2 - is approximately 100% of the sample absorbance.

      Spike 3 - is approximately 150% of the sample absorbance.

   d. The data for each MSA analysis will be identified in the raw data documentation along with the slope, intercept, and correlation coefficient (r) for the least squares fit of the data.

Reported values obtained by MSA will be flagged on the data sheet with the letter "S" if the correlation coefficient is greater than or equal to 0.995.

e. If the correlation coefficient (r) is less than 0.995, the MSA analysis will be repeated once. If the correlation coefficient is still less than 0.995, the result with the best correlation coefficient will be reported and flagged with a "+".

E. The above procedure is the procedure for CLP protocol. It is also further explained in Section E of your SOW 7/88 & 7/89. For Private Client protocol, ask your supervisor.

RAW DATA REQUIREMENTS

I. SAMPLE LABELING

A. All CLP Protocol must be labeled with EPA Sample Number and AATS I.D. Number.

B. All Non-CLP Protocol must be labeled with AATS I.D. and client name.

II. CODES FOR LABELING DATA

A. Table 1 located on page B-11 of SOW 7/88 is used to identify the following on all raw data:

1. Calibration standards, including source and preparation date.

2. Initial and Continuing Calibration Blanks and Preparation Blanks.

3. Initial and Continuing Calibration Verification standards, CRA standards.

4. Diluted and undiluted samples and all dilutions and volumes used to obtain the reported values.

5. Duplicates.

6. Spikes (indicating standard solutions used, final spike concentrations, and volumes used).

7. Instrument used, any instrument adjustments, data corrections or other apparent ab normalities in the measurement record, including all data voided or data not used to obtain reported values and a brief written explanation in the case narrative.

8. All information for each furnace analysis clearly and sequentially identified on the raw data:

   a. EPA Sample Number

   b. Sample and analytical spike data

   c. Percent recovery

   d. Coefficient of variation

e. Full MSA data:

- MSA correlation coefficient
- Slope
- Intercepts of linear fit
- Final sample concentration (standard addition concentration)
- Type of background correction used:

  BZ for Zeeman
  BS for Smith-Heiftje

*NOTE: For any samples on which MSA's are required, the samples will be labeled "0", "1", "2", or "3" at the end of the Sample I.D., with the value of the spike added by "1", "2", or "3".

9. Time and date of each analysis. Instrument run logs can be submitted if they contain this information.

   a. If the instrument does not automatically provide times of analysis, these must be manually entered on all raw data for Initial and Continuing Calibration Verifications and Blanks, as well as Calibration standards and CRDL standards.

10. Integration times for AA analysis.

LOGBOOK WRITE-UP

(Manually)

I. HEADER INFORMATION

A. Analyst: Fill in the analyst's initials, then your initials (JDR/JDR).

B. Date: Write in the day or days it was done on in the following format; mm/dd/yy.

C. Instrument: Fill in the instrument that the data was run on.

D. Bkgrd: Fill in the background correction used upon the analysis.

E. Element: Write in the element being analyzed.

F. Wavelength: Fill in the element's wavelength.

G. Calibration Source: Write in "Plasma-Chem STD 1A."

H. Calibration Prep Date: Fill in the first day of analysis.

II. CALIBRATION FOR ANALYSIS

A. Underneath the concentration column write in S###, with the ### standing for the concentration value for that standard.

B. Under the ABS column fill in the average absorbance for each standard (excluding S0 in which you would write in AZ).

1. If the analyst does happen to run more than 5 standards, then continue down into the samples box.

C. Under the r column (after you cross the "r" out and write in "Time") fill in the time for each analysis.

III. SAMPLES BOX

A. In the Client I.D. column should go the following:

1. EPA numbers

2. Private Client Numbers (if they are CLP Protocol)

3. Client Names (if Private Protocol)

*Note: The Private and EPA CLP protocol numbers at times will have/might have one of the following at the end of the number: D, S, 0, 1, 2, 3, or A.

B. In the AATS I.D. column should go the following:

1. QA/QC (ICV, ICB, CRA, CCV, CCB, PBS, PBW, LCSS, LCSW, BLK, BLK SPK);

2. AATS I.D.'s (which include all in the following format: ####.##)

*Note: If there is an MSA on the run, write in the AATS number for the first one, then write in on the spiked samples the value it was spiked at.

C. The first Burn ABS/Conc. column should be used for the times of each analysis. Cross out the heading and write "Time" above the box. For PE5100 runs, the times should be on the raw data and for the TJA22 and PE5000, the times should be written in manually.

D. The second Burn ABS/Conc. column is used only for the absorbance of MSA's. Write in the absorbance only. The concentrations are not needed for MSA's. Circle the "ABS" in the heading when using this column.

E. In the Average ABS/Conc. column should be written all of the average concentrations exactly how they are shown on the raw data.

F. The Dilution Factor column is used for designating dilutions. For example, a sample that was diluted 1:4 will have a final concentration X 5. Write the dilution factor in for both the straight sample and the analytical sample.

G. The Final Concentration column should be used for all the final calculated data. For diluted samples, be sure to multiply by the correct factor. For CLP Protocol samples under the IDL, write in the IDL in this column. For Non-CLP Protocol samples under the CRDL, write in the CRDL. For example: a CLP Protocol sample has a value of 0.80. The IDL is 2.0, therefore, the value written in is 2.0U. The same applies for Non-CLP Protocol. When rounding up numbers, use the following rule: less than 10 = 2

significant figures; greater than 10 = 3 significant figures.

H. The Comments column is used for writing the following:

1. True Value/Percent Recovery: for ICV, CCV, matrix and analytical spikes.

2. MSA: If the sample requires one.

3. HRSD(S) or (A): with the % RSD between injections written above. (S) stands for the sample and (A) stands for the analytical sample.

4. L%R: If the percent recovery of the analytical spike is <40%.

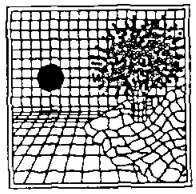
5. Overcal: This is for samples higher than our largest standard.

6. Dil X:X: to call for a dilution next time we run that sample.

7. Any explanation about the performance of the instrument or the order of samples.

IV. MULTI-PAGE WRITE-UPS

A. When going onto the next page with the same calibration, the calibration box does not have to be written up again. Only fill out the header information as detailed in Section I, A-H. This applies to all pages under the same calibration.



FURNACE RUNLOG

AATS / SWOK

WAVELENGTH:			INTEGRATION TIME:	INSTRUMENT:	ANALYST/DATE:
Arsenic	=	193.70	5.0 Sec.		
Lead	=	283.30			
Selenium	=	196.00	BKGRND CORRECTION:	DATA FILE:	ELEMENT RAN:
Thallium	=	276.00			AS PB SE TL

#	Lab ID #	Client ID #	Used	Used
1.				
2.				
3.				
4.				
5.				
6.				
7.				
8.				
9.				
10.				
11.				
12.				
13.				
14.				
15.				
16.				
17.				
18.				
19.				
20.				

#	Lab ID #	Client ID #	Used	Used
21.				
22.				
23.				
24.				
25.				
26.				
27.				
28.				
29.				
30.				
31.				
32.				
33.				
34.				
35.				
36.				
37.				
38.				
39.				
40.				

AUTOMATIC DATA WRITE-UPS

I. PERKIN-ELMER 5100s

A. Before starting the analysis, make sure you have put in your data file of XYYYYZZ;

1. X is either A or B depending on which instrument is used.

2. Y is the month and day.

3. Z is the hour the run is started.

B. The next step is to reformat the files.

1. Enter "Reformat Files" in windows selection.

2. Select the file desired to be reformatted from the selection on the screen and press "Enter".

3. From the list of selections, select Smartlog.

4. After you have typed out the appropriate date, file select "Execute Reformat".

C. Now to copy the file to the diskette:

1. Exit to DOS

2. Go into the \AA_USER\AA_FILES data directory.

3. Type "COPY XYYYYZZ.PRN A:"

D. The next step is to transfer it to the Telecations software. After you reach the Smartlog Program on another computer:

1. Load the Furnace file

a. Go to the "Load File" option and hit "Enter".

b. Choose "F5" for an archive file.

c. Type "FURNACE". You are now in the \ARCHIVES\FURNACE directory.

2. Load the data file

   a. Cursor to "Automatic" and hit "Enter".

   b. Choose "F5" for ASCII file.

   c. Type "A:XYYYYZZ.PRN", and press "Enter".

   d. Type "8;1;16;57;40;17;41;0" to choose the fields, press "Enter", and press "F10" to load.

E. Choose "Data Review" from the main menu, and press "F2" to review all data. You will need to make sure everything is entered correctly and in accordance with the manual write-up requirements.

F. To type out the run, after updating the data, you will need to isolate the run;

   1. Choose "Data Review"

      a. Isolate the data by "Report Name"

      b. Type in the given report name

      c. When the isolated data comes up on the screen, press "F8" for "Print Report"

      d. You would then press "F6" for "Custom"

      e. Then type in the correct report name and press "Enter.

G. As for now the TJA22 and PE5000 do not have the capabilities for automatic write-ups, but are expected soon.

   • Procedures may change due to the process of an upcoming network.

   • This is a short summery covering the automatic data entry. If any further information is needed, refer to "Logbook Write-Up - Manually".

WORKSHEET REQUIREMENTS

I. Worksheets should be finished by the instruments that started them under the same linear range.

A. When starting a new worksheet, always look on the prepped furnace case sheet on the clipboard to find the case which is due first. Always start the case which is due the soonest, unless told otherwise.

1. When a sample is finished:

a. Write your initials and the date in the Date/Analyst column. If the entire case worked, with no reruns, put an arrow all the way down the column. If there are any reruns, adjust arrows accordingly.

b. The Result column is the actual concentration, rounded to necessary significant figures. The IDL or CRDL is also written in this column. When a figure is written in this column, this signifies that the sample has been done.

c. The Comments column is used to write the instrument used, as well as rerun comments.

The following initials are used for instruments:

PE5100A - A
PE5100B - B
PE5000 - P
TJA22 - T

The following comments are to be written in case of reruns:

HRSD(S) - write %RSD above when RSD of sample is >20%
HRSD(A) - write %RSD above when RSD of analytical sample is >20%
L %R - when analytical recovery is <40%
MSA - write concentration of sample above
H dil (X:X) - be sure to write in dilution needed when concentration is above calibration curve.

B. Attach worksheet to raw data and logbook write-up and place in appropriate box for data review.

KEEPING OF BOOKS

I. MAINTENANCE LOGBOOK

A. Maintenance Logbooks are kept to record any adjustments, instrument repair, part replacement, etc. The analyst should also note any problems or anything they might perceive to be a problem with the instrument in the future. After the entry, the analyst is required to initial and date it.

II. PARAMETERS LOGBOOK

A. This book is designed to keep track of the most current optimized conditions of the HGA programs. When a HGA program is changed, it is up to the analyst to place the new parameters into the parameters book under the correct instrument, and date the most current one.

III. CALIBRATION STANDARDS

A. The procedure of how and when each standard, ICV, modifier needs to be made is in the front of the Calibration and Stock Standards book.

B. The "Preparation Dates of Furnace Calibration Standards and Modifiers" sheet is pretty much self explanatory. At the top of the sheet is a place for all the Log numbers of the chemicals used. Each sheet is designed for a week of prep, the date of each day goes on top of the ═══ entry. The initials of the analyst who did the prep goes on the line corresponding with the date and standard done.

IV. INVENTORY BOOK

A. The purpose of this book is to keep track of all the parts we use on our instruments to make sure we have the necessary backup supplies. The three sheets are the following:

1. Parts List: to be filled out for each instrument or instruments if similar. These should be updated every two weeks and then the sheet dated and signed.

2. Running Inventory: the sheet is kept to show when we replaced or used a part on one of the instruments.

3. Lamp Log: is kept for keeping track of each lamp as we receive them. This is done in case we can return it under warranty for replacement. So it is important for the analyst to record all information on the Lamp Log that there is room for.

MERCURY ANALYSIS IN WATER BY MANUAL COLD VAPOR

I. Application

A. Tested concentration range: 0.2 to 10.0 ug/l.

B. Approximate instrument detection limit: 0.15 ug/l.

C. Interferences:

1. Sulfide - eliminated by the addition of potassium permanganate.

II. Apparatus

A. Instrumentation

1. Instrumentation Laboratories Model 357 Atomic Absorption Spectrophotometer.

2. Instrumentation Laboratories Model 440 Atomic Vapor Accessory.

3. Mercury hollow cathode lamp.

4. Zero-grade argon.

5. Absorption cell: Standard quartz spectrophotometer cell, 10 cm long with open ends.

6. Aeration tubing: Tygon tubing for passage of the mercury vapor from the sample bottle to the absorption cell.

B. Reagents

1. Sulfuric acid, conc.: Baker Instra-Analyzed for trace metals analysis.

2. Sulfuric acid, 0.5 N: Dilute 14.0 ml of conc. sulfuric acid to 1.0 liter.

3. Nitric acid, conc.: Baker Instra-Analyzed for trace metals analysis.

4. Hydrochloric acid, conc.: Baker Instra-Analyzed for trace metals analysis.

5. Stannous chloride: Add 25 grams stannous chloride to 500 ml 25% HCl.

6. Sodium chloride - hydroxylamine hydrochloride solution: Dissolve 12 grams of NaCl and 12 grams hydroxylamine hydrochloride in distilled water and dilute to 100 ml.

7. Potassium permanganate, 5% solution, w/v: Dissolve 5 grams of potassium permanganate in 100 ml of distilled water.

8. Potassium persulfate, 5% solution, w/v: Dissolve 5 grams of potassium persulfate in 100 ml of distilled water.

C. Parameters

1. Wavelength: 253.3 nm

2. Lamp current: 3.0

3. Bandpass: 1.0 nm

4. Readout mode: Peak height

5. Integration time: 8.0 seconds

6. Background correction: D_2

III. Procedure

A. Standard Preparation

1. Stock solutions for mercury are prepared solutions from Spex Industries containing 1,000 mg mercury/l.

2. An intermediate stock solution of 10.0 mg/l mercury is prepared by diluting 1.0 ml of stock solution and 1.0 ml of conc. HNO_3 to 100 ml with ASTM Type II water. This standard is prepared weekly.

3. A second intermediate solution of 0.1 mg/l mercury is prepared each day of analysis by diluting 1.0 ml of the 10.0 mg/l intermediate stock solution and 1.0 ml of conc. HNO_3 to 100 ml

with ASTM Type II water. Make fresh daily.

4. Calibration standards are prepared by diluting the 0.1 mg/l intermediate standard to 100 ml with ASTM Type II water according to the following schedule:

Standard Calibration ug/l	ml of 0.1 mg/l Intermediate Standard
0.0	0.0
0.5	0.5
1.0	1.0
5.0	5.0
10.0	10.0

5. Working calibration standards are prepared fresh each day of analysis and carried through the digestion procedure outlined in Section III.B.

B. Sample Preparation

1. Add the standards prepared above in Section III.A.4. to 300 ml fleaker.

2. Transfer 100 ml of well mixed sample to a 300 ml fleaker.

3. Add 5 ml of conc. sulfuric acid and 2.5 ml of conc. nitric acid to each bottle.

4. Add 15 ml of $KMnO_4$ solution to each bottle and allow to stand at least 15 minutes.

5. Add 8 ml of potassium persulfate to each bottle.

6. Heat for 2 hours in a water bath at 95°C.

7. Cool and add 6 ml of sodium chloride-hydroxylamine sulfate solution to reduce the excess permanganate.

C. Calibration

1. Instrument Calibration

a. Set up instrument according to manufacturers recommendations.

b. Attach the stannous chloride to the Atomic Vapor Accessory

and cycle through several times to insure that stannous chloride is being pumped to the sample chamber.

c. Pour the blank sample into a 150 ml fleaker and attach to the IL Atomic Vapor Accessory.

d. Start the cycle and establish the baseline.

e. Beginning with the blank and working towards the highest standard, run the solutions and record the peak height readings.

2. Analysis of Calibration Data

a. Tabulate the calibration standard concentration versus peak height response for each calibration standard.

b. Analyze data by linear regression on a calculator. The correlation coefficient must be greater than 0.995 before proceeding with analysis.

3. Calibration Checks

a. Initial Calibration Verification

- After the system has been calibrated, the accuracy of the initial calibration shall be verified and documented by the analysis of an EPA supplied standard carried through the digestion procedure.

- The measurement must be within $\pm$ 20% of the true value or the analysis must be terminated, the problem corrected, the instrument recalibrated and the calibration reverified.

b. Calibration Blank

- A calibration blank is analyzed at the beginning and end of the run and at a frequency of 10% during the run.

- Blanks are to be reported down to the instrument detection limit.

- If the blank result is greater than 0.2 ppb, terminate analysis, correct the problem and recalibrate.

c. Continuing Calibration Verification

- To assure calibration accuracy during each analysis run, an independently prepared standard solution carried through the digestion procedure will be analyzed at a frequency of 10% and after the last analytical samples.

- The mercury concentrations will be at the mid-range level of the calibration curve.

- If the deviation of the continuing calibration verification is greater than 20%, the instrument will be recalibrated and the proceeding samples since the last calibration verification will be reanalyzed.

D. Instrument Analysis

1. Set up instrument parameters as discussed in Section II.C.

2. Calibrate instrument as discussed in Section III.C.1.

3. Add samples to Atomic Vapor Accessory and record peak height responses.

4. Using the calibration curve generated in Section III.2., convert peak height responses to concentration.

5. Report mercury concentrations as follows:
   Below 0.2 ug/l = 0.2 u
   Between 0.2 and 10 ug/l = one decimal place
   Above 10 ug/l = whole numbers

IV. Quality Control

A. Initial calibration verification: Analyzed immediately after instrument calibration. Control limits are ± 20% of the true value. (Supplied by EPA).

B. Initial calibration blank: Analyzed immediately after initial calibration verification. Control limits are ± 0.2 ug/l.

C. Continuing calibration verification: Analyzed at a frequency of 10% or every two hours during an analysis run, whichever is more frequent. Control limits are 80% to 120% of the true value.

D. Continuing calibration blank: Analyzed at a frequency of 10% or every two hours during an analysis run, whichever is more frequent. Control limits are ± 0.2 ppb.

E. Preparation blank: At least one preparation blank must be prepared and analyzed with every Sample Delivery Group or with each batch of samples digested, whichever is more frequent.

F. Matrix spike sample: At least one spike analysis is performed on each group of samples of a similar matrix type and concentration or for each Sample Delivery Group, whichever is more frequent. Samples identified as field blanks cannot be used for spiked sample analysis.

G. Post-digested spike: When the matrix spike sample recovery falls outside the control limits of 75% to 125% and the sample concentration does not exceed 4 times the spike added, a post-digested spike is performed. Spike the unspiked aliquot of the sample at 2 times the indigenous level or 2 times CRDL, whichever is greater.

H. Duplicate sample: One duplicate sample is analyzed from each group of samples of a similar matrix type and concentration or for each Sample Delivery Group, whichever is more frequent. Samples identified as field blanks cannot be used for duplicate sample analysis. A control limit of 20% for relative percent difference (RPD) is used for original and duplicate sample values greater than or equal to 5 times CRDL. A control limit of ± CRDL is used for sample values less than 5 times CRDL.

I. Laboratory control sample: An aqueous LCS is not required for accuracy.

J. Holding times: The holding time for mercury is 21 days from verified time of sample receipt to sample digestion.

MERCURY ANALYSIS IN SOIL BY MANUAL COLD VAPOR

I. Application

A. Tested concentration range: 0.1 - 5.0 mg/kg.

B. Approximate detection limit: 0.08 mg/kg.

C. Interferences:

1. Sulfide - eliminated by the addition of potassium permanganate.

II. Apparatus

A. Instrumentation

1. Instrumentation Laboratories Model 357 Atomic Absorption Spectrophotometer.

2. Instrumentation Laboratories Model 440 Atomic Vapor Accessory.

3. Mercury hollow cathode lamp.

4. Zero-grade argon.

5. Absorption cell: Standard quartz spectrophotometer cell, 10 cm long with open ends.

6. Aeration tubing: Tygon tubing for passage of the mercury vapor from the sample to the absorption cell.

B. Reagents

1. Sulfuric acid, conc.: Baker Instra-Analyzed for trace metals analysis.

2. Sulfuric acid, 0.5 N: Dilute 14.0 ml of conc. sulfuric acid to 1.0 liter.

3. Nitric acid, conc.: Baker Instra-Analyzed for trace metals analysis.

4. Hydrochloric acid, conc.: Baker Instra-Analyzed for trace metals analysis.

5. Stannous chloride: Add 25 grams stannous chloride to 500 ml 25% HCl.

6. Sodium chloride - hydroxylamine hydrochloride solution: Dissolve 12 grams of NaCl and 12 grams hydroxylamine hydrochloride in distilled water and dilute to 100 ml.

7. Potassium permanganate, 5% solution, w/v: Dissolve 5 grams of potassium permanganate in 100 ml of distilled water.

8. Potassium persulfate, 5% solution, w/v: Dissolve 5 grams of potassium persulfate in 100 ml of distilled water.

C. Parameters

1. Wavelength: 253.3 nm

2. Lamp current: 3.0

3. Bandpass: 1.0 nm

4. Readout mode: Peak height

5. Integration time: 8.0 seconds

6. Background correction: D_2

III. Procedure

A. Standard Preparation

1. Stock solutions for mercury are prepared solutions from Spex Industries containing 1,000 mg mercury/l.

2. An intermediate stock solution of 10.0 mg/l mercury is prepared by diluting 1.0 ml of stock solution and 1.0 ml of conc. HNO_3 to 100 ml with ASTM Type II water. This standard is prepared weekly.

3. A second intermediate solution of 0.1 mg/l mercury is prepared each day of analysis by diluting 1.0 ml of the 10.0 mg/l intermediate stock solution and 1.0 ml of conc. HNO_3 to 100 ml

with ASTM Type II water. Make fresh daily.

4. Calibration standards are prepared by diluting the 0.1 mg/l intermediate standard to 100 ml with ASTM Type II water according to the following schedule:

Standard Calibration ug/l	ml of 0.1 mg/l Intermediate Standard
0.0	0.0
0.5	0.5
1.0	1.0
5.0	5.0
10.0	10.0

5. Working calibration standards are prepared fresh each day of analysis and carried through the digestion procedure outlined in Section III.B.

B. Sample Preparation

1. Add the standards prepared above in Section III.A.4. to 300 ml fleaker.

2. Weigh a representative 0.2 gram portion of wet sample and place in a 300 ml fleaker.

3. Add 5.0 ml of conc. sulfuric acid and 2.5 ml of conc. nitric acid to each bottle.

4. Heat 2 minutes in a water bath at 95°C.

5. Cool, add 50 ml distilled water, 15 ml potassium permanganete and 8 ml of potassium persulfate solution.

6. Mix thoroughly and place in the water bath for 30 minutes at 95°C.

7. Cool and add 6 ml of sodium chloride-hydroxylalmine sulfate.

8. Add 55 ml of distilled water.

C. Calibration

1. Instrument Calibration

a. Set up instrument according to manufacturers recommendations.

b. Attach the stannous chloride to the Atomic Vapor Accessory and cycle through several times to insure that stannous chloride is being pumped to the sample chamber.

c. Pour the blank sample into a 150 ml fleaker and attach to the IL Atomic Vapor Accessory.

d. Start the cycle and establish the baseline.

e. Beginning with the blank and working towards the highest standard, run the solutions and record the peak height readings.

2. Analysis of Calibration Data

a. Tabulate the calibration standard concentration versus peak height response for each calibration standard.

b. Analyze data by linear regression on a calculator. The correlation coefficient must be greater than 0.995 before proceeding with analysis.

3. Calibration Checks

a. Initial Calibration Verification

- After the system has been calibrated, the accuracy of the initial calibration shall be verified and documented by the analysis of an EPA supplied standard carried through the digestion procedure.

- The measurement must be within $\pm$ 20% of the true value or the analysis must be terminated, the problem corrected, the instrument recalibrated and the calibration reverified.

b. Calibration Blank

- A calibration blank is analyzed at the beginning and end of the run and at a frequency of 10% during the run.

- Blanks are to be reported down to the instrument detection limit.

- If the blank result is greater than 0.2 ppb, terminate analysis, correct the problem and recalibrate.

c. Continuing Calibration Verification

- To assure calibration accuracy during each analysis run, an independently prepared standard solution carried through the digestion procedure will be analyzed at a frequency of 10% and after the last analytical samples.

- The mercury concentrations will be at the mid-range level of the calibration curve.

- If the deviation of the continuing calibration verification is greater than 20%, the instrument will be recalibrated and the proceeding samples since the last calibration verification will be reanalyzed.

D. Instrument Analysis

1. Set up instrument parameters as discussed in Section II.C.

2. Calibrate instrument as discussed in Section III.C.1.

3. Add samples to Atomic Vapor Accessory and record peak height responses.

4. Using the calibration curve generated in Section III.2., convert peak height responses to concentration.

5. Convert concentrations to dry weight by the following:

$$\text{Concentration, ug/g} = \frac{C \times V}{W \times S}$$

Where: C = concentration , ug/l
V = final volume in liters after sample preparation
W = weight in grams of wet sample in preparation
S = percent solids/100

IV. Quality Control

A. Initial calibration verification: Analyzed immediately after instrument calibration. Control limits are ± 20% of the true value. (Supplied by EPA).

B. Initial calibration blank: Analyzed immediately after initial calibration verification. Control limits are ± 0.2 ug/l.

C. Continuing calibration verification: Analyzed at a frequency of 10% or every two hours during an analysis run, whichever is more frequent. Control limits are 80% to 120% of the true value.

D. Continuing calibration blank: Analyzed at a frequency of 10% or every two hours during an analysis run, whichever is more frequent. Control limits are ± 0.2 ppb.

E. Preparation blank: At least one preparation blank must be prepared and analyzed with every Sample Delivery Group or with each batch of samples digested, whichever is more frequent.

F. Matrix spike sample: At least one spike analysis is performed on each group of samples of a similar matrix type and concentration or for each Sample Delivery Group, whichever is more frequent. Samples identified as field blanks cannot be used for spiked sample analysis.

G. Post-digested spike: When the matrix spike sample recovery falls outside the control limits of 75% to 125% and the sample concentration does not exceed 4 times the spike added, a post-digested spike is performed. Spike the unspiked aliquot of the sample at 2 times the indigenous level or 2 times CRDL, whichever is greater.

H. Duplicate sample: One duplicate sample is analyzed from each group of samples of a similar matrix type and concentration or for each Sample Delivery Group, whichever is more frequent. Samples identified as field blanks cannot be used for duplicate sample analysis. A control limit of 20% for relative percent difference (RPD) is used for original and duplicate sample values greater than or equal to 5 times CRDL. A control limit of ± CRDL is used for sample values less than 5 times CRDL.

I. Laboratory control sample: The EPA-provided solid LCS is prepared and analyzed using the sample procedures as the soil samples. If the

result for the solid LCS falls outside the control limits established by EPA, the analyses must be terminated, the problem corrected and the samples associated with the LCS redigested and reanalyzed.

J. Holding times: The EPA-CLP holding time for mercury is 21 days from verified time of sample receipt to sample digestion.

MERCURY RUN LOG

Calibration			
Std.#	Conc.	Abs.	r
Std.0			
Std.1			
Std.2			
Std.3			
Std.4			

Analyst_____ Date_____

Instrument_____ Bkgrd_____

Element_____ Wavelength_____

Calibration Standard: Source_____ Prep Date_____

Client I.D.	SWLO I.D.	Time of Analysis	wt/vol	Percent ABS	Dilution Factor	Final Concentration ug/l mg/kg	Comments

SWLO FORM 1027W (12/89)

075

RESIDUE, FILTERABLE

Method 160.1 (Gravimetric, Dried at 180°C)

STORET NO. 70300

1. Scope and Application
   - 1.1 This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes.
   - 1.2 The practical range of the determination is 10 mg/l to 20,000 mg/l.
2. Summary of Method
   - 2.1 A well-mixed sample is filtered through a standard glass fiber filter. The filtrate is evaporated and dried to constant weight at 180°C.
   - 2.2 If Residue, Non-Filterable is being determined, the filtrate from that method may be used for Residue, Filterable.
3. Definitions
   - 3.1 Filterable residue is defined as those solids capable of passing through a glass fiber filter and dried to constant weight at 180°C.
4. Sample Handling and Preservation
   - 4.1 Preservation of the sample is not practical; analysis should begin as soon as possible. Refrigeration or icing to 4°C, to minimize microbiological decomposition of solids, is recommended.
5. Interferences
   - 5.1 Highly mineralized waters containing significant concentrations of calcium, magnesium, chloride and/or sulfate may be hygroscopic and will require prolonged drying, desiccation and rapid weighing.
   - 5.2 Samples containing high concentrations of bicarbonate will require careful and possibly prolonged drying at 180°C to insure that all the bicarbonate is converted to carbonate.
   - 5.3 Too much residue in the evaporating dish will crust over and entrap water that will not be driven off during drying. Total residue should be limited to about 200 mg.
6. Apparatus
   - 6.1 Glass fiber filter discs, 4.7 cm or 2.1 cm, without organic binder, Reeve Angel type 934-AH, Gelman type A/E, or equivalent.
   - 6.2 Filter holder, membrane filter funnel or Gooch crucible adapter.
   - 6.3 Suction flask, 500 ml.
   - 6.4 Gooch crucibles, 25 ml (if 2.1 cm filter is used).
   - 6.5 Evaporating dishes, porcelain, 100 ml volume. (Vycor or platinum dishes may be substituted).
   - 6.6 Steam bath.
   - 6.7 Drying oven, 180°C ±2°C.
   - 6.8 Desiccator

Approved for NPDES
Issued 1971

6.9 Analytical balance, capable of weighing to 0.1 mg.

7. Procedure

7.1 Preparation of glass fiber filter disc: Place the disc on the membrane filter apparatus or insert into bottom of a suitable Gooch crucible. While vacuum is applied, wash the disc with three successive 20 ml volumes of distilled water. Remove all traces of water by continuing to apply vacuum after water has passed through. Discard washings.

7.2 Preparation of evaporating dishes: If Volatile Residue is also to be measured heat the clean dish to 550 ±50°C for one hour in a muffle furnace. If only Filterable Residue is to be measured heat the clean dish to 180 ±2°C for one hour. Cool in desiccator and store until needed. Weigh immediately before use.

7.3 Assemble the filtering apparatus and begin suction. Shake the sample vigorously and rapidly transfer 100 ml to the funnel by means of a 100 ml graduated cylinder. If total filterable residue is low, a larger volume may be filtered.

7.4 Filter the sample through the glass fiber filter, rinse with three 10 ml portions of distilled water and continue to apply vacuum for about 3 minutes after filtration is complete to remove as much water as possible.

7.5 Transfer 100 ml (or a larger volume) of the filtrate to a weighed evaporating dish and evaporate to dryness on a steam bath.

7.6 Dry the evaporated sample for at least one hour at 180 ±2°C. Cool in a desiccator and weigh. Repeat the drying cycle until a constant weight is obtained or until weight loss is less than 0.5 mg.

8. Calculation

8.1 Calculate filterable residue as follows:

$$\text{Filterable residue, mg/l} = \frac{(A - B) \times 1{,}000}{C}$$

where:

A = weight of dried residue + dish in mg
B = weight of dish in mg
C = volume of sample used in ml

9. Precision and Accuracy

9.1 Precision and accuracy are not available at this time.

Bibliography

1. Standard Methods for the Examination of Water and Wastewater, 14th Edition, p 92, Method 208B, (1975).

SOUTHWEST LABORATORY OF OKLAHOMA, INC.

STANDARD OPERATING PROCEDURE FOR THE ANALYSIS OF TOTAL RECOVERABLE PETROLEUM HYDROCARBONS

IN210

REV 1.0 —3/6/91

Summary: Samples are extracted with trichlorotrifluoroethane (freon) to remove petroleum hydrocarbons. Silica gel is added to the extract to remove interferences. Petroleum hydrocarbons in the extract are then determined by infrared analysis.

PROCEDURE:

I.WATER SAMPLES

A. SEPARATORY FUNNEL TECHNIQUE

1. Acidify the 1 liter sample to pH <2 with HCl. Decant the entire sample into a 2 liter separatory funnel. Add 30 ml freon to the sample bottle and shake vigorously to remove hydrocarbons remaining on the container. Vent the container frequently to prevent pressure buildup. Add this freon to the separatory funnel.
2. Vigorously shake the separatory funnel for two minutes to thoroughly mix the two phases, venting as necessary to prevent pressure buildup. Allow the phases to separate.
3. Filter the freon phase through enough sodium sulfate to resolve any emulsion and remove entrained water.
4. Repeat steps 2 and 3 twice more using 30 additional ml freon each time.
5. After filtering the last of the freon extract through the sodium sulfate, rinse the separatory funnel tip and the sodium sulfate twice to remove traces of hydrocarbon.
6. Accurately measure the sample volume.
7. Bring the extract volume to 100 ml. Stir in 3 g silica gel and measure the absorbance using an infrared spectrophotometer at 2930 cm^{-1}.
8. Total Recoverable Petroleum Hydrocarbons are calculated as follows:

$$C_{TPH} = (A_{sample}/A_{standard})(C_{standard})(100/V)$$

Where: C= concentration (mg/l)
A= absorbance
V= sample volume (ml)

II. SOIL/SLUDGE SAMPLES

A. SOXHLET EXTRACTOR TECHNIQUE

1. Weigh out 1-30 grams (depending on the expected level of contamination) of sample into an extraction thimble.
2. Stir in anhydrous sodium sulfate and mix until the mixture is of uniform consistency and free-flowing.
3. Place thimble in a clean soxhlet extractorand extract for 4 hours with freon. Cycle rate should be maintained at 20 cycles/hour.

4. Bring the extract volume to 100 ml. Stir in 3 g silica gel and measure the absorbance using an infrared spectrophotometer at 2930 cm^{-1}.
5. Total Recoverable Petroleum Hydrocarbons are calculated as follows:

$$C_{TPH} = (A_{sample}/A_{standard})(C_{standard})(100/W)$$

Where: $C_{standard}$ = concentration (mg/l)
C_{TPH} = concentration (mg/kg)
A = absorbance
W = sample weight (g)

B. SONICATION TECHNIQUE

1. Weigh 15-30 grams of sample into a clean 400ml beaker.
2. Stir in anhydrous sodium sulfate and mix until the mixture is of uniform consistency and free-flowing.
3. Add sufficient freon to completely cover the sample and allow immersion of the sonicator probe tip one-fourth inch into the liquid.
4. Immerse the probe tip into the liquid (1/8 to 1/4 inch). Sonicate three minutes at 50-80% duty cycle and 7-9 output control.
5. Decant the freon extract through sodium sulfate and collect.
6. Repeat steps 3-5 twice more.
4. Bring the extract volume to 100 ml. Stir in 3 g silica gel and measure the absorbance using an infrared spectrophotometer at 2930 cm^{-1}.
5. Total Recoverable Petroleum Hydrocarbons are calculated as follows:

$$C_{TPH} = (A_{sample}/A_{standard})(C_{standard})(100/W)$$

Where: $C_{standard}$ = concentration (mg/l)
C_{TPH} = concentration (mg/kg)
A = absorbance
W = sample weight (g)

C. "MISSOURI" METHOD

1. Weigh out 30g of sample.
2. Stir in anhydrous sodium sulfate and mix until the mixture is of uniform consistency and free-flowing.
3. Pack the sample into a chromatography column.
4. Elute 100 ml freon through the packed column and collect the eluent.
5. Stir in 3g silica gel and measure the absorbance using an infrared spectrometer at 2930 cm^{-1}.
6. Total Recoverable Petroleum Hydrocarbons are calculated as follows:

$$C_{TPH} = (A_{sample}/A_{standard})(C_{standard})(100/W)$$

Where: $C_{standard}$ = concentration (mg/l)
C_{TPH} = concentration (mg/kg)
A = absorbance
W = sample weight (g)

APPENDIX B

Site Health and Safety Forms

30 180

Routing:	
Safety Engineer	

Safety Training Session Report

Division	Project Location *(if applicable)*	Department or Group
Date of Meeting	Duration *(not to exceed 20 minutes)*	Total Personnel in Attendance

Safety Items Discussed:

Roster of Attendees:

Signed _____
Responsible Supervisor

FIELD SAFETY CHECKLIST

Work Location: _____

Date

1. Reviewed work plans with client representative: _____

2. Requested maps of aboveground and underground utilities: _____

3. Reviewed utility maps: _____
   (water supply, firewater, sewer, process sewer,
   electric, gas, telephone, other underground piping)

4. Met with utility representative to review utility locations and asked each utility the following questions:

   a. Any underground utilities at work site location?
   b. Any on-going construction that would affect field activities?
   c. Any vapor releases associated with unit operations?
   d. Any other hazards associated with operating units?
   e. Any special requirements?

   (Names and name of utility: _____)

5. Determined if any permits required: _____
   Type: _____

6. Obtained necessary permits: _____
   Permit expiration date: _____

7. Request MSDS for any on-site chemicals: _____

8. Client's established monitoring protocol if any: _____

9. Obtained final approval for commencement of work: _____

Comments:

AGREEMENT AND ACKNOWLEDGEMENT STATEMENT

Site Safety Plan Agreement

Burns & McDonnell's Project Manager or Site Health and Safety Supervisor personnel have the authority to stop any work performed by Burns and McDonnell's subcontractors, if any work is not performed in accordance with the requirements of this Site Health and Safety Plan.

All Burns & McDonnell project personnel and subcontractor personnel are required to sign the following agreement prior to performing work at the site.

1. I have read and fully understand the Site Safety Plan and my individual responsibilities.

2. I agree to abide by the provisions of the Site Safety Plan.

Name	Signature
Company	Date
Name	Signature
Company	Date
Name	Signature
Company	Date
Name	Signature
Company	Date

Name	Signature
Company	Date
Name	Signature
Company	Date
Name	Signature
Company	Date
Name	Signature
Company	Date
Name	Signature
Company	Date
Name	Signature
Company	Date

HEALTH AND SAFETY PLAN FIELD AMENDMENT FORM

Project Name: _____

Project Number: _____

Location: _____

Changes in field activities or hazards:

Proposed Amendment:

Proposed By: _____ Date: _____
Site Health and Safety Supervisor
or Others

Approved By: _____ Date: _____
Project Manager

_____ Date: _____
Health & Safety Department Manager

Declined By: _____ Date: _____

Amendment Number: _____

Amendment Effective Date: _____